A CRYOSTAT FOR THERMAL CONDUCTIVITY MEASUREMENTS AT LOW TEMPERATURE

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A CRYOSTAT FOR THERMAL CONDUCTIVITY
MEASUREMENTS AT LOW TEMPERATURE

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SUMMARY

This work describes the design and construction of a cryostat for measuring the thermal conductivity of metals and alloys between 20° and 150°K. The cryostat described is, in general, of the same basic type used by previous workers in this field. A detailed description of the construction and operation of the cryostat and its associated vacuum systems and electrical systems is given.

The primary function of the cryostat described in this work is to measure the temperature at two points a known distance apart on the specimen and the power supplied to the specimen. From these measurements and the geometry of the specimen, the mean thermal conductivity of the specimen is calculated at an average temperature. The following table shows the mean thermal conductivity of a free-machining yellow brass as determined in this work. The nominal composition of this brass is 62 per cent copper, 35 per cent zinc, and 3 per cent lead.

Mean Thermal Conductivity of Yellow Brass

Average Temperature, °K	Mean Thermal Conductivity, watts/cm - °K
84.67	0.486
88.73	0.498
98.90	0.528
109.01	0.564
118.24	0.605

These data when plotted fitted a straight line drawn through the data to within two per cent. This is considered to be well within the overall accuracy of the data.

Wright (4) has also made thermal conductivity measurements for this same brass specimen in a cryostat of somewhat different design. A comparison of the results obtained with the two cryostats showed that Wright's thermal conductivity data are approximately ten per cent lower over the range of these data.

A secondary function of the cryostat is to calibrate the thermometric elements used in the thermal conductivity experiments. Two copper-constantan thermocouples were calibrated against a platinum resistance thermometer. The operation of the cryostat as a thermometer calibrator and the results of the calibration experiments are discussed. The calibration data for the thermocouples were fitted to the expression:

$$E = 5879.7 - 5.3973\,T - 0.060976\,T^2 + 3.02 \times 10^{-6}\,T^3$$

where E is the e.m.f. in microvolts and T is the temperature in °K. This expression fitted the experimental data to within 0.08 per cent, and may be expected to give temperatures to within ± 0.15 degree in the range 60° to 150°K. Using this expression an e.m.f.-temperature table was constructed which gives e.m.f. values for each degree of temperature.

At the same time that calibration experiments were made for the thermocouples, two carbon resistance thermometers were also calibrated. These carbon thermometers were made of commercially available carbon resistors (Ohmite, 39 ohms, 1 watt, silver band). The results of the calibration of the carbon thermometers indicated that cooling the thermometers from room temperature to 78°K affected the resistance of the thermometers, but these variations in resistance appeared to be eliminated after cooling from room temperature several times. Thus only one calibration point was obtained for the carbon resistance thermometers. The results of the experiments at this point indicated that the resistance of these thermometers was reproducible to ± 0.05 ohm (± 0.15 degree) at a temperature of 78.25°K.

CHAPTER I

INTRODUCTION

Within recent years cryogenic engineering has become recognized as a new branch of engineering and not merely a branch of refrigerating engineering. The growth of this new field of engineering is a result of the increasing use of very low temperatures and low temperature refrigerants in science and industry. This rapid growth of cryogenic engineering has presented many problems to the engineer and scientist responsible for the design of equipment to produce and maintain these very low temperatures and to handle and store low temperature liquids and solids. The problem may, in part, be attributed to a lack of accurate and extensive data on the electrical, mechanical, and thermal properties of materials of construction. A knowledge of the thermal conductivity of materials is essential for the adequate design of equipment for use at very low temperatures. With this knowledge, materials of construction can be selected which ensure minimum transfer of heat, and thus simplify the problem of isolating and maintaining these very low temperatures.

In 1954, three extensive surveys of the thermal conductivity data for metals and alloys at low temperatures were made by Powell and Blanpied (1), Powell (2, 3), and Wright (4). An examination of these reviews indicates wide, unexplored regions between room temperature and absolute zero where thermal conductivity data do not exist.

The purpose of the present work was to design and construct a cryostat for measuring the thermal conductivity of metals and alloys at low temperatures. Such a cryostat has been constructed for use in the range 20° to 150°K. A detailed description of the construction of the cryostat and the associated vacuum systems and the electrical circuits is given in Chapters II and III. The cryostat was used to calibrate two copper-constantan thermocouples by comparison with a calibrated platinum resistance thermometer. These thermocouples were used to measure temperature in later thermal conductivity measurements. The operation of the cryostat as a thermometer calibrator, and the results of these experiments are discussed in Chapter IV. As part of the calibration experiments, two carbon resistance thermometers were also calibrated. These results are reported in Appendix D. Finally, the cryostat was used to measure the thermal conductivity of a specimen of free-machining yellow brass, a material widely used in the construction of low temperature equipment. The results of these experiments are discussed in Chapter V.

Measurement of thermal conductivity.--Thermal conductivity is defined by Fourier's law for conduction of heat through a solid and is expressed mathematically for one-dimensional heat flow by the relation:[1]

$$\frac{dq}{d\theta} = -k\ A\ \frac{dt}{dx} \qquad (1)$$

where dq is the amount of heat flowing in the differential time $d\theta$; A is the area of the section at right angles to the direction of heat flow;

[1] McAdams (5).

$-\frac{dt}{dx}$ is the temperature gradient or the rate of change of temperature (t) with respect to the length of the path (x); and k is a proportionality factor known as the thermal conductivity. To be of practical use in determining thermal conductivity, certain assumptions must be made which permit the integration and simplification of this relation.

If steady-state heat flow is assumed, the amount of heat flowing is independent of time. Thus, $\frac{q}{\theta}$ or Q can be substituted for $\frac{dq}{d\theta}$, where Q is the amount of heat flowing per unit of time. If it is also assumed that the area of the section at right angles to the direction of heat flow is constant, the area is independent of the length of the path. When the variation of thermal conductivity with temperature is known, these two assumptions permit the integration of equation (1).

The effect of temperature on the thermal conductivity of metals and alloys varies. For alloys the thermal conductivity generally decreases with a decrease in temperature. This decrease is often essentially linear over the range from room temperature to about 100°K. For pure metals the thermal conductivity is less predictable, and is found to increase and decrease with a decrease in temperature depending upon the purity, degree of strain, etc. For example, copper has a thermal conductivity of 4.1 watts/cm-°K at 150°K, 13.0 watts/cm-°K at 30°K, and 10.5 watts/cm-°K at 23°K.[2] Many pure or slightly impure metals exhibit such a maximum in thermal conductivity at low temperatures. No simple relation is, therefore, available which relates the thermal conductivity of relatively pure metals with temperature over

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[2]Data of Powers, Schwartz, and Johnston (1951) from Powell and Blanpied (1).

the complete range of temperature. However, over sufficiently small temperature intervals it is usually acceptable to assume that the thermal conductivity varies linearly with temperature even for pure metals.

These three assumptions, namely steady-state process of heat flow, constant area at right angles to the heat flow path, and thermal conductivity varies linearly with temperature between small increments of temperature, permit the integration of equation (1) between the limits of t_1 and t_2 and x_1 and x_2. Equation (1) can be thus integrated to:

$$k_m = -\frac{Q}{A\,\frac{(t_2 - t_1)}{(x_2 - x_1)}} = -\frac{Q}{A\,\frac{\Delta t}{\Delta x}} \tag{2}$$

where Δt and Δx are substituted for $t_2 - t_1$ and $x_2 - x_1$ and k_m is the thermal conductivity at the mean temperature $t_1 + t_2/2$. Of the three factors on which thermal conductivity is dependent, the area is usually the most readily obtained. The other factors, rate of heat flow and temperature difference between two points in the heat flow path, are the data which are determined in a thermal conductivity cryostat.

Experimental technique.--In 1953, Olsen and Rosenberg (6) published an article on thermal conductivity of metals at low temperatures. In this paper, a brief discussion of the experimental techniques used to determine the thermal conductivity of metals and alloys was given. The following discussion on experimental technique is based primarily on this article.

In general, the basic designs of cryostats for determining the thermal conductivity of metals are very similar. The specimen to be studied is usually in rod form and is mounted vertically in an evacuated container to eliminate heat transfer by conduction and convection to the walls of the container. One end of the specimen is in either direct or indirect thermal contact with a suitable fixed-temperature bath, such as liquid hydrogen, nitrogen, etc. The selection of this bath depends on the temperature range in which measurements are to be made. A temperature measuring device, e.g. a thermometer, is fitted at one or more points along the specimen to measure the temperature or the temperature gradient. To the free end of the specimen, a small electrical heater is fitted to supply heat. This heater has potential and current leads to permit measurement of the power supplied. At higher temperatures heat losses by radiation are appreciable, and a radiation shield is provided to decrease the magnitude of these losses. This shield encloses the specimen, heater, and thermometer.

The magnitude of the temperature difference between two points on the specimen for which the thermal conductivity is being determined may vary from 0.01 degree to several degrees. The magnitude of the difference used is dependent on the temperature region in which experimental work is done. For example, if the temperature region is the liquid helium range, small temperature differences are used, because the temperature range (1.8° - 5°K) is small and difficult to obtain and maintain. If the temperature region is the liquid nitrogen range (55° - 150°K), larger temperature differences are frequently used.

Gas thermometers, electrical resistance thermometers, and thermocouples are used as thermometric elements to measure either an absolute temperature or a temperature gradient, the choice being determined by the temperature region and the accuracy required in the temperature measurement. Certain advantages are associated with the use of each of these thermometric elements. Electrical resistance thermometers and thermocouples have the advantage of requiring readily available external measuring equipment -- a potentiometer or a bridge. They are simpler to make and attach than are gas thermometers, and where there are spacial limitations, are generally less bulky than gas thermometers. However, gas thermometers are widely used because they are unaffected by a magnetic field; thus, they are useful in the study of superconductors and magneto-resistive effects. Another advantage of gas thermometers is that the ideal gas laws can be assumed if the gas pressure is not too high, thus fewer calibrations points are required. Aside from the fact that gas thermometers are usually more bulky than resistance thermometers and thermocouples, another disadvantage of gas thermometers is that a correction for the external volume of the system is required. However, this correction can be made small by keeping the external volume sufficiently small.

When liquid refrigerants can be employed, steady temperatures are maintained by allowing the refrigerant to boil under reduced, atmospheric, and high pressures up to the critical pressure. This is easily accomplished, but does not provide thermal conductivity data over the complete range of temperature between liquid helium and room temperature. The liquid refrigerants used for maintaining temperatures between liquid helium and room temperatures include helium, hydrogen, nitrogen, solid carbon dioxide and acetone, and ice water. To maintain steady temperatures at intermediate temperatures between the refrigerant ranges, two methods are used. The first method is to allow the refrigerant gas to expand slowly and by controlling this expansion to maintain a steady temperature. The second method is a controlled "heat leak" between the specimen and a liquid refrigerant bath. In this method, the specimen is attached to a block on which an electrical heater is wound. This block is connected thermally to a refrigerant bath by a small wire, usually copper. By supplying power to the block heater, the temperature of the block can be maintained at any temperature above the bath temperature by establishing an equilibrium between the heat flowing into the block from the heater and the heat flowing from the block to the refrigerant bath. The "heat leak" method is more satisfactory for temperatures above the critical point of the refrigerant, because of the time required to obtain equilibrium conditions for poor conductors.

It is difficult to obtain perfect thermal isolation of the specimen within the cryostat; as a result, corrections must be made for certain heat losses (or gains). These heat losses vary in cause and magnitude and include losses due to radiation and to conduction along heater and thermometer leads. Radiation losses in the liquid hydrogen region are negligible, but are significant above about 60°K. To decrease the effect of radiation in the higher temperature ranges, radiation shields wound with a heater are used. This permits setting the temperature of the shield at approximately the same temperature as the specimen and reduces the effect of radiation. Heat losses by conduction along leads presents a more difficult problem. These losses can be minimized by placing the leads in good thermal contact with the refrigerant bath; however, a correction for this heat loss is always required.

<u>The cryostat</u>.--The cryostat used in this work was designed for use in determining the thermal conductivity of metals and alloys between 20° and 150°K. In general, it is the same basic arrangement described in the preceding section of this chapter. Because of the ease of fabrication and operation and the availability of external equipment, copper-constantan thermocouples were used to measure the temperature at two points along the specimen. Such thermocouples were used by Tyler and Wilson (7) for temperature measurements between liquid hydrogen and room temperature. Carbon resistance thermometers, made of commercial carbon resistors, were also used for measuring temperatures. Such resistors have been used by Olsen and Renton (8) below one degree Kelvin and by Clement and Quinell (9) up to the liquid hydrogen range. The selection of these resistance thermometers in the present work is a result of an interest in their use at temperatures above the liquid hydrogen range.

The cryostat was designed to use liquid hydrogen and nitrogen for maintaining the desired temperatures. These refrigerants are fairly standard and used by many investigators. Time did not permit any actual measurements with liquid hydrogen.

The method of controlled "heat leak" was used to maintain temperatures at intermediate temperatures between the refrigerant ranges. This method was used extensively by Powers, Schwartz, and Johnston (10) and by

Tyler and Wilson (7) in the range of liquid hydrogen to room temperature. This method of establishing intermediate temperatures was selected because it permits faster and easier operation.

The primary function of this cryostat was to measure the temperature at two points a known distance apart on the specimen and the power supplied to the specimen. From the resulting measurements and the geometry of the specimen, the mean thermal conductivity of the specimen was calculated at an average temperature. Secondary functions which can be performed in the cryostat are (1) calibration of thermometric elements, (2) use as a calorimeter for the measurement of specific heat between 20° and 150°K, and (3) use for the study of the cooling effects resulting from the desorption of gas from solid adsorbents. All of the external equipment for these functions are not described, but provisions for carrying out these additional experiments were considered in the design of the equipment. The details of the construction of the cryostat are discussed in Chapter II.

CHAPTER II

CONSTRUCTION OF CRYOSTAT

In this chapter a discussion is given of the construction of the cryostat, the vacuum systems, and the electrical systems. This discussion includes the details of how each part was fabricated and assembled.

Cryostat.--A schematic diagram of the cryostat which was constructed is shown in Figure 1. The principal features of the cryostat can be seen by referring to this diagram. These features are (1) the refrigerant pot assembly, including the refrigerant pot P and the associated tubes leading through the supporting plate AA; (2) the controlled heat leak assembly, consisting of the copper plate EE and associated control heater H1, attached to the bottom of the refrigerant pot by the copper wire heat leak F and supporting rods N; and (3) the specimen assembly which includes the specimen S and its associated thermocouples and heater. These three assemblies are surrounded by a metal vacuum case J attached to the plate AA. In normal operation the cryostat is immersed in liquid nitrogen to a depth approximately two inches above the plate AA, while a high vacuum is maintained in the vacuum case. The desired refrigerant, usually liquid nitrogen, is placed in the pot P.

Figure 2 gives a more detailed view of the controlled heat leak and specimen assemblies. Figure 3 is a photograph of the assembled

Legend for Figure 1

AA	Top of Vacuum Case
BB	Top of Charcoal Can
CC	Top of Refrigeration Pot
DD	Bottom of Charcoal Can and Refrigerant Pot
EE	Copper Plate
F	Copper Wire Heat Leak
H1	Copper Plate Heater
H2	Monel Radiation Shield Heater
HN1	Refrigerant Pot Heater
HT	Specimen Heater
J	Thermal Conductivity Cryostat Vacuum Case
K	Charcoal Can
L	Calorimeter Ring
N	Copper Plate Support Rods
P	Refrigerant Pot
R1	Copper Radiation Shield
R2	Monel Radiation Shield
S	Specimen
T1, T2	Carbon Resistance Thermometers
TC1, TC2, TC3, TC4	Copper-Constantan Thermocouples
TD1, TD2	Copper-Constantan Difference Couples
TS	Platinum Resistance Thermometer and Well
U	Refrigerant Pot Vent Tube
V1	Thermal Conductivity Cryostat Wire Seal and Vacuum Tube
V2	Calorimeter Vacuum Tube
V3	Refrigerant Pot Filling and Vacuum Tube
V4	Charcoal Can Filling and Vacuum Tube
W1	Thermal Conductivity Cryostat Wire Seal Tube
W2	Calorimeter Wire Seal Tube
X	Extra Tube
Y	Water Well
Z	Gas Thermometer Capillary Exit Tube

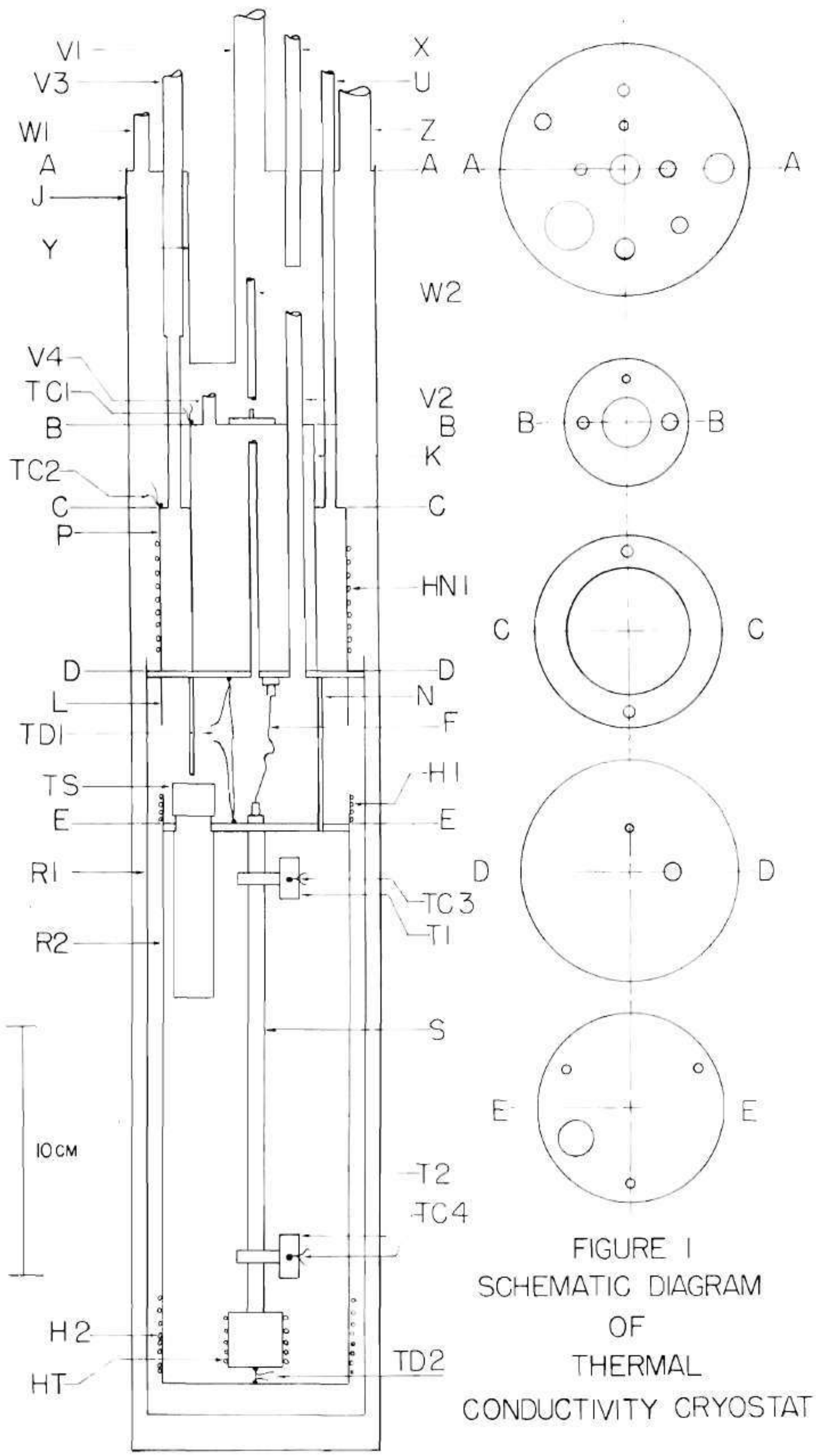

FIGURE I
SCHEMATIC DIAGRAM
OF
THERMAL
CONDUCTIVITY CRYOSTAT

Legend for Figure 2

F	Copper Wire Heat Leak
H1	Copper Plate Heater
H2	Monel Radiation Shield Heater
HT	Specimen Heater
K	Charcoal Can
L	Calorimeter Ring
N	Copper Plate Support Rods
P	Refrigerant Pot
R1	Copper Radiation Shield
R2	Monel Radiation Shield
S	Specimen
TC3, TC4	Copper-Constantan Thermocouples
TD1, TD2	Copper-Constantan Difference Couples
TS	Platinum Resistance Thermometer and Well
V2	Calorimeter Vacuum Tube
W2	Calorimeter Wire Seal Tube

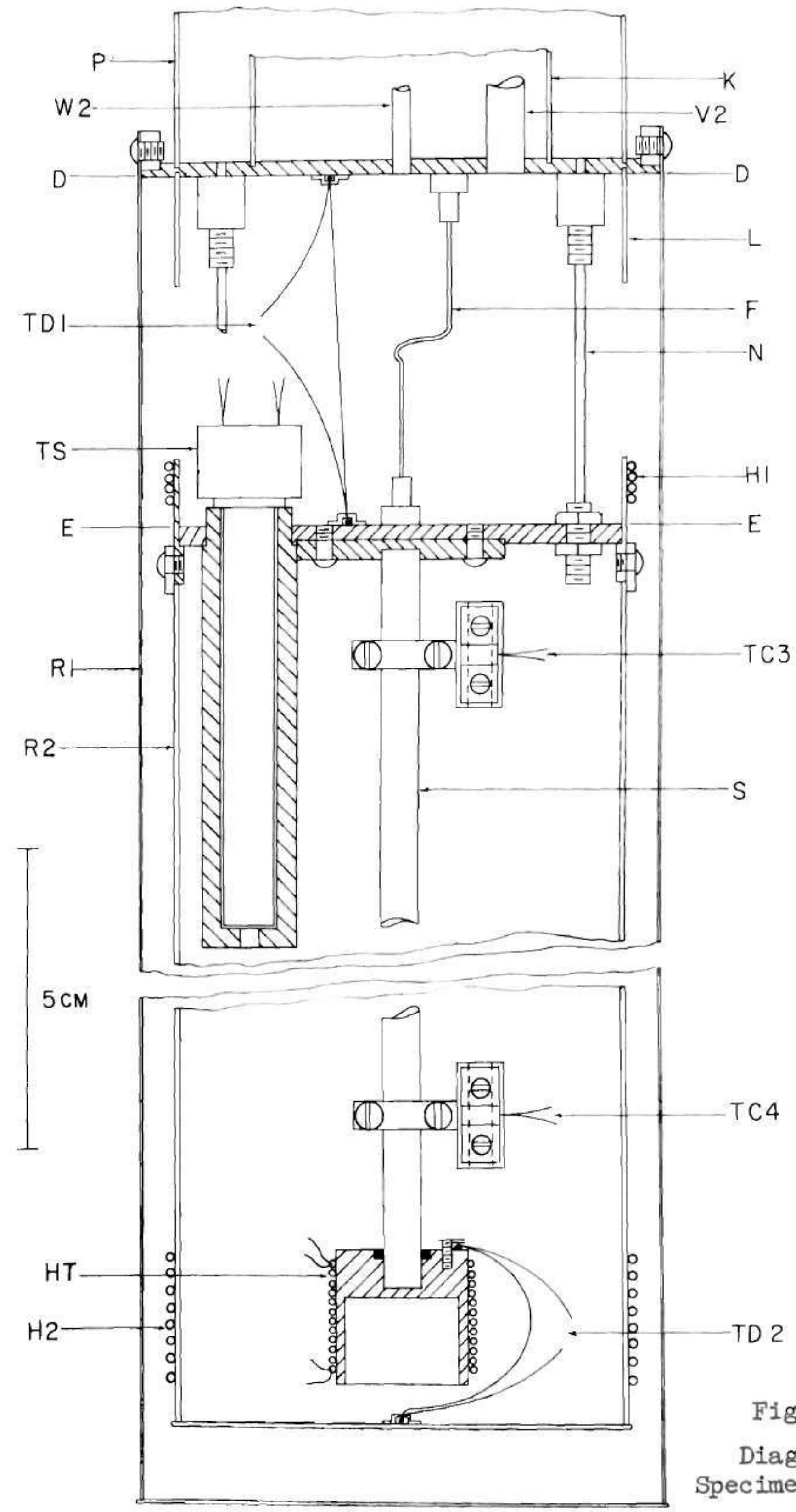

Figure 2

Diagram of
Specimen Assembly

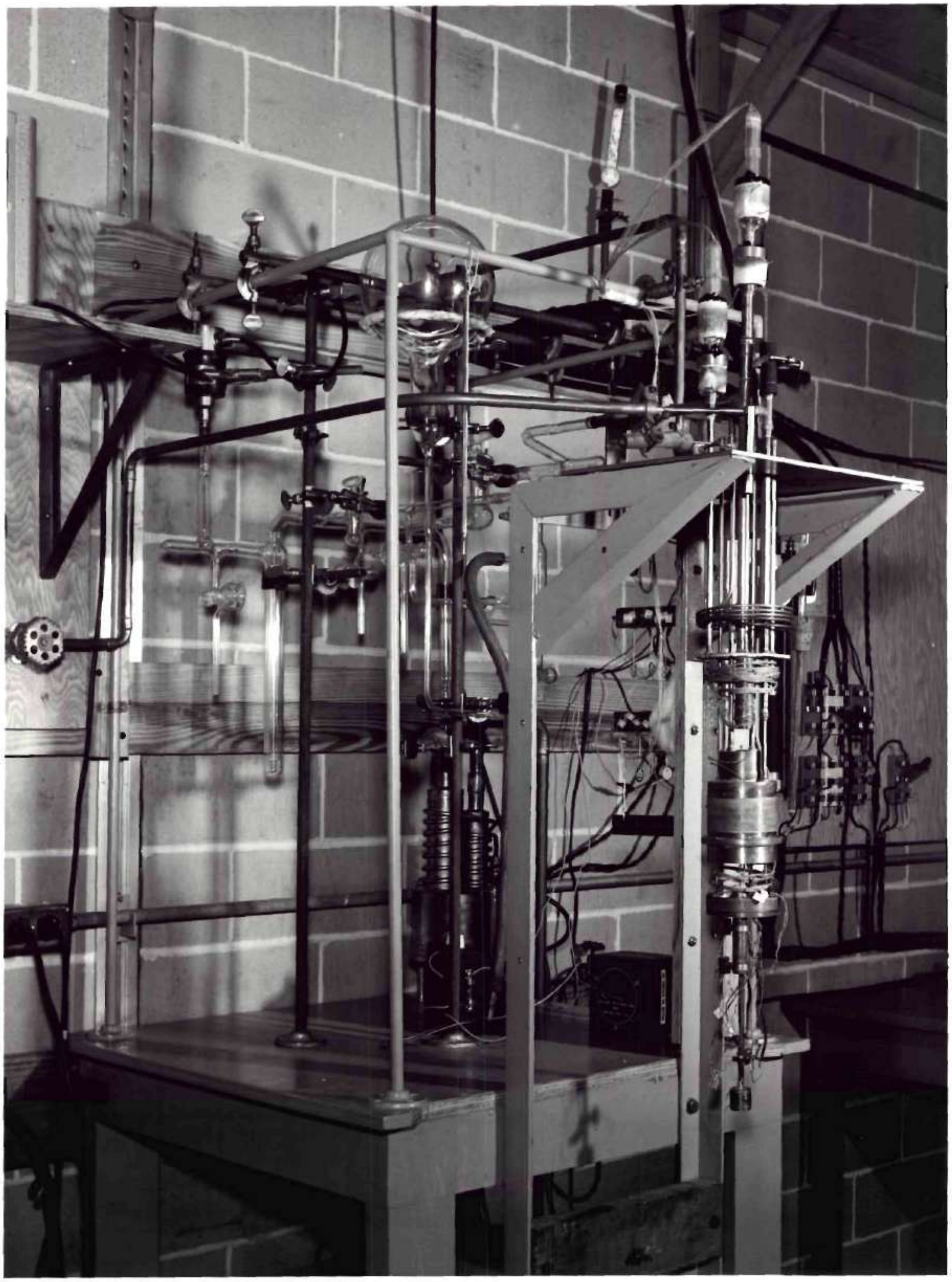

Figure 3
Thermal Conductivity Cryostat

cryostat and associated vacuum system with the vacuum case removed. The dimensions and weights of the cryostat parts are given in Table 1 of Appendix A.

The details of the specimen assembly which includes the specimen S and its associated thermocouples TC3 and TC4 and heater HT are shown in Figure 2. The thermocouples TC3 and TC4 were attached to the specimen by small copper clamps. The heater HT was wound on a copper shell and was soldered with Wood's metal to one end of the specimen. To the other end of the specimen a copper disc was soldered with Wood's metal. This disc was then attached with screws to the copper plate EE, a part of the controlled heat leak assembly. Two sets of clamps, heater shells, and discs were provided, one set for a 1/4 inch diameter specimen and one for a 3/8 inch diameter specimen.

The specimen assembly was surrounded by the radiation shield R2 which was also attached with screws to the copper plate EE. The cylindrical section of this shield was made of monel and wrapped with a single layer of aluminum foil. The bottom of the shield was made of brass and silver soldered to the cylindrical section. The heater H2 was wound around the bottom of the shield, so that a temperature gradient could be established along the shield similar to the gradient along the specimen. Control of the gradient was maintained by the difference couple TD2, which measured the temperature difference between the radiation shield and the specimen heater.

The controlled heat leak assembly which includes the copper plate EE and associated control heater H1 is attached to the bottom of the refrigerant pot by the copper wire heat leak F and supporting rods N as shown in Figure 2. The copper plate was soldered inside the brass ring on which the control heater H1 was wrapped. To the copper plate was also soldered the copper well in which the platinum resistance thermometer TS was mounted. The copper plate and control heater were attached to the bottom of the refrigerant pot P (plate DD) by the heat leak wire F and the support rods N. The heat leak wire was a B&S No. 16 gauge copper wire about 3 inches long. To each end of the wire was soldered a small brass fixture which was soldered to the copper plate EE and the brass plate DD. This wire was the principal "heat leak" between the plates in addition to supporting the copper plate and control heater. The support rods N for the copper plate EE were made of copper-nickel tubes (1 millimeter diameter and 0.010 inch wall thickness) and were 2 3/4 inches long. Brass screws were soldered to each end of these tubes, so that the plate EE and rods could be removed from the cryostat.

The controlled heat leak assembly was surrounded by the radiation shield R1. This shield was attached with screws to a brass ring which was soldered to the brass plate DD. The shield was made of copper sheet metal and lined on both sides with a single layer of aluminum foil.

The refrigerant pot assembly includes the refrigerant pot P, the charcoal can K, and the associated control heater HN1 and tubes leading through the supporting plate AA. This assembly is shown in Figure 1. The refrigerant pot P was made of brass, and all joints were silver soldered. The volume of the pot was 160 cc. Around the pot was wrapped the heater HN1 to permit the evaporation of excess refrigerant. The temperature was measured by the thermocouple TC2, which was soldered directly to the top of the pot. The pot was filled with refrigerant through the tube V3 and vented through the tube U.

The charcoal can K was made of brass, and all joints were silver soldered. The volume of this can was 185 cc. Soldered on the top of this can was a 3/4 inch diameter manhole cover, which could be removed to fill the can with the adsorbent on which adsorption experiments were to be run. The temperature was measured by the thermocouple TC1 soldered to the top of the can. The gas to be adsorbed entered the can through the tube V4. Desorption of the gas was accomplished by reducing the pressure with a mechanical vacuum pump, which was also connected to the tube V4.

To the bottom of the charcoal can and refrigerant pot was silver soldered the brass ring L. This ring was provided as a means of attaching a calorimeter vacuum case.

The tubes associated with the refrigerant pot assembly were used to support the assembly. These tubes pass through the plate AA and extend through another brass plate from which the cryostat was rigidly suspended from the angle iron frame. (This can be seen in Figure 3.) All tubes were fitted with brass ferrules and silver soldered to the plate AA and the brass plate. A list of the tubes, which gives the size, material, and use of each tube, can be found in Table 2, Appendix A.

The brass vacuum case J surrounded the complete cryostat and was soft soldered to the plate AA. This case could contain a high vacuum (1×10^{-6} mm Hg).

Vacuum system.--The high vacuum system was connected to the thermal conductivity cryostat by an "O" ring seal attached to the tube V1 and by a metal-to-glass seal to the tube V2. The necessary high vacuum was obtained by using an oil diffusion pump (Consolidated Vacuum Corp. VMF - 20w) backed by a good mechanical pump (Welch Mfg. Co. Duo-Seal Model 1400 B). A vacuum of approximately 1×10^{-6} mm Hg was regularly obtained with this arrangement. The vacuum was measured with a Phillips Gauge Type PHG-09, which was calibrated by the manufacturers to an accuracy of $\pm$ 10 per cent over the range of the instrument (0.5 to 10^{-7} mm Hg). Provisions were also made in the glass vacuum system to store and admit helium gas into the cryostat for heat exchange purposes.

A second vacuum pumping system was provided to reduce the pressure in the refrigerant pot through the tube V3. This system contained a mechanical vacuum pump (Duo-Seal Model 1400 B, rated at 1×10^{-3} mm Hg) and was constructed of 1/2 inch o.d. soft copper tubing. Sweat copper fittings were used, and all joints were soft soldered. The pressure was measured with a 0-30 inch Bourdon gauge.

Electrical system.--The electrical leads entered the cryostat through two Lucite plugs, which were sealed with Apiezon wax W and mounted on the tubes, V1 and W1. The insulation was removed from the wires before sealing. These seals were modeled after one described by Johnston and Kerr (11). A total of 50 wires -- fourteen of these wires were spares -- pass through the plugs. Inside the cryostat the wires were wrapped around the water well (Y) several times to protect them during the sealing of the vacuum case. Several additional loose turns of wire were made in the cryostat before the leads were connected. This decreased the effect of the heat leak along the leads. Outside of the cryostat, the electrical leads were attached to junction blocks to which connections from the control and measuring instruments were made.

A complete list of the electrical leads in the cryostat is given in Table 3, Appendix A. This table gives the number, size, kind of wire, and the use of the leads.

All heaters in the cryostat were made of double nylon, enameled constantan wire with single cotton, enameled copper wire leads. After winding, the heaters were given a generous coating of GE adhesive No. 7031 (two parts adhesive to one part thinner). The adhesive was then allowed to air dry at room temperature. A description of the heaters is given in Table 4, Appendix A.

The difference couples, TD1 and TD2, were single junction couples, made from B&S No. 34 gauge, single cotton, enameled copper wire and B&S No. 32 gauge, double nylon, enameled constantan wire. This wire came from the same spools that Wright (4) used to prepare his calibrated difference couples. After twisting a copper and constantan wire together, the junction was soldered, insulated with Cellophane tape, and wrapped in a piece of thin copper sheet metal. The junctions were then clamped into position by small copper bridges.

Four single junction thermocouples, TC1, TC2, TC3, and TC4, were used in the cryostat. Each thermocouple junction was made from the same copper and constantan wire that was used to make the difference couples. The junctions of the thermocouples (TC3 and TC4) were prepared in the same manner described above for the difference couples. These thermocouples were used to measure the temperature at points along the specimen and were clamped to the specimen by means of specially designed copper clamps. The calibration of these thermocouples is described in Chapter IV. The junction of the thermocouples (TC1 and TC2) were soldered directly to the charcoal can and refrigerant pot as shown in Figure 1. These couples were not calibrated since they were only used for control purposes.

The reference junction of each thermocouple (TC1, TC2, TC3, and TC4), located outside the cryostat, was placed in a separate 3 mm o.d. pyrex tube filled with Nujol (heavy mineral oil). The four tubes were then wrapped together and immersed in a 10 mm i.d. pyrex tube also filled with Nujol. This larger tube was then immersed in a crushed ice-water mixture which served as the reference temperature.

The carbon resistance thermometers were made from commercially available carbon resistors (Ohmite, 39 ohm, 1 watt, silver band) taken from stock. To mount the resistors, the plastic covering was first removed by grinding. The "bare" resistor was coated, first, with a thin layer of GE adhesive No. 7031 (two parts adhesive to one part thinner), then a double layer of cigarette paper, and finally another coat of adhesive. After drying, each resistor was sealed in a copper block with adhesive and allowed to dry at room temperature. Two leads of B&S No. 34 gauge, single cotton, enameled copper wire were soldered about 1/2 inch from each end of the resistor. The use of two leads made possible the measurement of the current and potential through the resistors. The calibration data obtained for the carbon thermometers is discussed in Appendix D.

CHAPTER III

ELECTRICAL CIRCUITS

The construction of the heaters and thermometric elements in the cryostat have been discussed in Chapter II. This chapter describes the components of the electrical circuits and their operation for current and potential measurements of the heaters and thermometric elements. The instruments used in the circuits along with pertinent data on these instruments are listed in Table 5, Appendix A.

Potentiometer circuit.--Potential measurements across the specimen heater, standard resistors, and thermometric elements were made with a Leeds and Northrup, Type K-2 potentiometer in combination with either of two Leeds and Northrup galvanometers. The potentiometer was not calibrated in an absolute sense but was certified by the manufacturer to be accurate to 0.02 per cent. When the potential unbalance was unknown or quite large, a pointer type galvanometer was used with the potentiometer to adjust the circuits into approximate balance. A high-sensitivity galvanometer was used with the potentiometer to make the final adjustments necessary to balance the circuits. Power was supplied to the potentiometer circuit by a 2-volt, 100 amp-hour, low discharge Willard battery. An Eppley standard cell was used with the potentiometer as a reference for the potential measurements. This cell was calibrated by the manufacturer and reported to have a voltage of 1.01879 international volts (1.01926 absolute volts) at 25°C.

<u>Specimen heater circuit</u>.--The components of the specimen heater circuit necessary to operate and to measure the potential and current of the heater (160 ohms) are shown in Figure 4. The heater shown in this circuit is for use with a 1/4 inch diameter specimen, but another heater is available that can be used with a 3/8 inch diameter specimen. The power input to the heater was supplied by a 14-cell Edison nickel-iron-caustic battery (terminal voltage of 16 volts). The current through the heater circuit was regulated and controlled by using the variable resistance (R_v), which was made up of two variable resistors in series. One resistor was a Leeds and Northrup resistance box (0-9999 ohms), and the other was a Biddle rheostat (896 ohms). To assist in regulating and controlling the current, an ammeter[1] (0-100 milliamperes) was connected in the circuit to insure that the current did not exceed 100 milliamperes. The current through the heater circuit was determined by measuring the potential across the Leeds and Northrup standard resistance. This standard resistance was calibrated by the National Bureau of Standards in November, 1952 and found to have a resistance of 1.00044 absolute ohms at 25°C. Potential measurements across the heater were measured with the Type K-2 potentiometer as discussed previously. It was necessary to use a Leeds and Northrup volt box to aid in this potential measurement, since the potentiometer could measure a maximum voltage of 1.61 volts. This volt box had settings of 1 to 200 and was certified by the National Bureau of Standards in April, 1950 to be correct within 0.01 per cent. The timer in this circuit was a Haydon electric clock. The time could be read to the nearest five seconds and was considered to be accurate

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[1]Not shown in Figure 4.

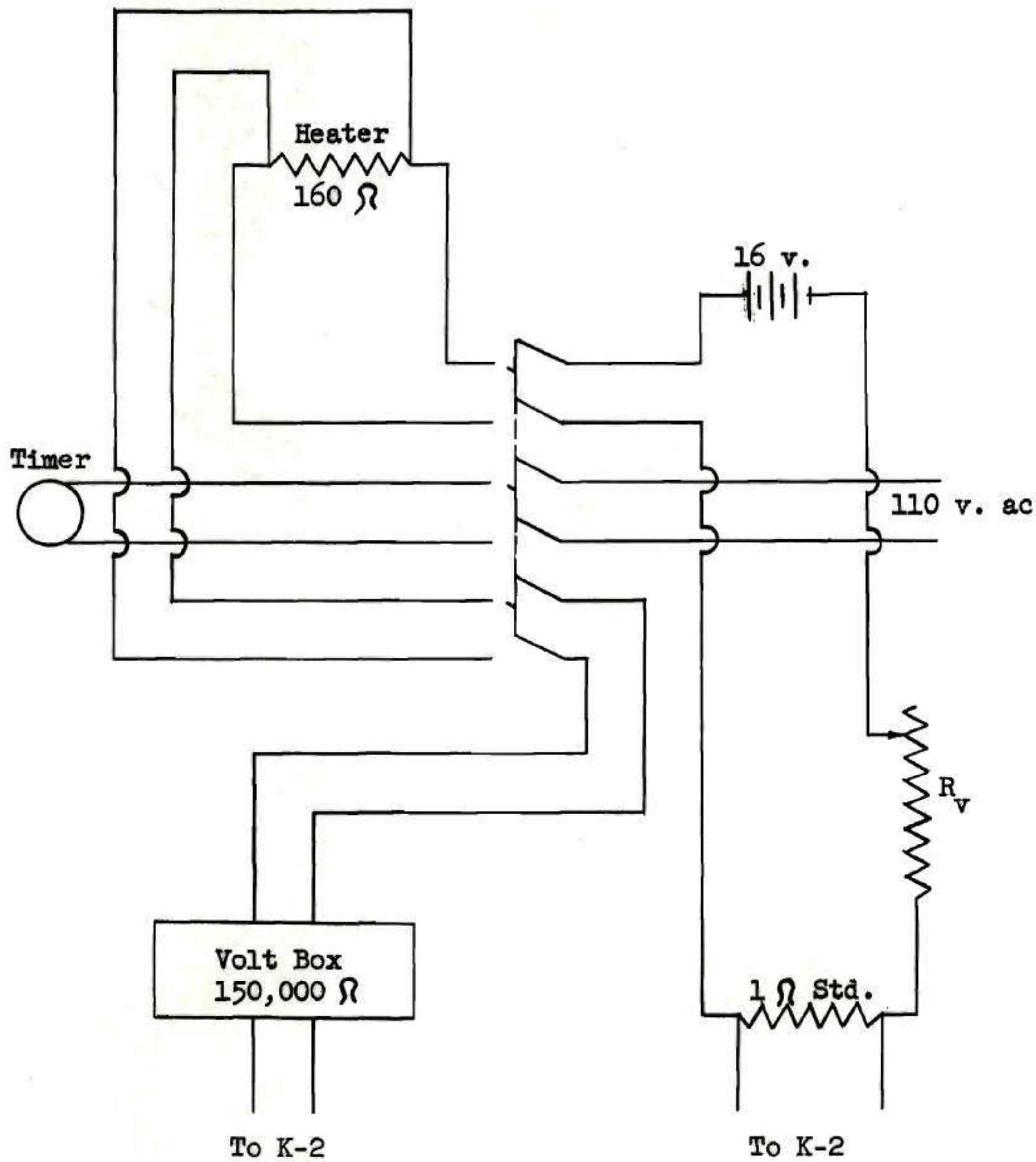

Figure 4

Specimen Heater Circuit

enough for these measurements. Potential measurements across the heater and standard resistor were made as a function of time in order to investigate the rate of heating and the change in current.

<u>Carbon resistance thermometer circuit</u>.--The carbon resistance thermometers (T1 and T2), made of commercially available carbon resistors, have an approximate resistance of 45 and 46 ohms, respectively, at 26°C. The circuit necessary for the determination of the resistance of these thermometers is shown in Figure 5.

The current through the circuit was supplied by a 2-volt, 100 amp-hour, low discharge Willard battery and was set at approximately one milliampere by a Rubicon resistance box (1800 ohms). The current through the circuit was determined by measuring the potential across a 100-ohm Rubicon standard resistor. This standard resistor was certified by the manufacturer in June, 1949 to have a resistance of 100.002 absolute ohms and to be accurate to 0.01 per cent. The potential across the carbon thermometers and the standard resistor were measured with the K-2 potentiometer and the high sensitivity galvanometer as previously discussed. These potential measurements were also measured as a function of time.

<u>Secondary heater circuits</u>.--Figure 6 is a diagram of the secondary heater circuits. The secondary heaters are so designated because they are used for control purposes in the cryostat. There are three of these heaters; the refrigerant pot heater (HN1), the copper plate control heater (H1), and the monel radiation shield heater (H2). The diagram

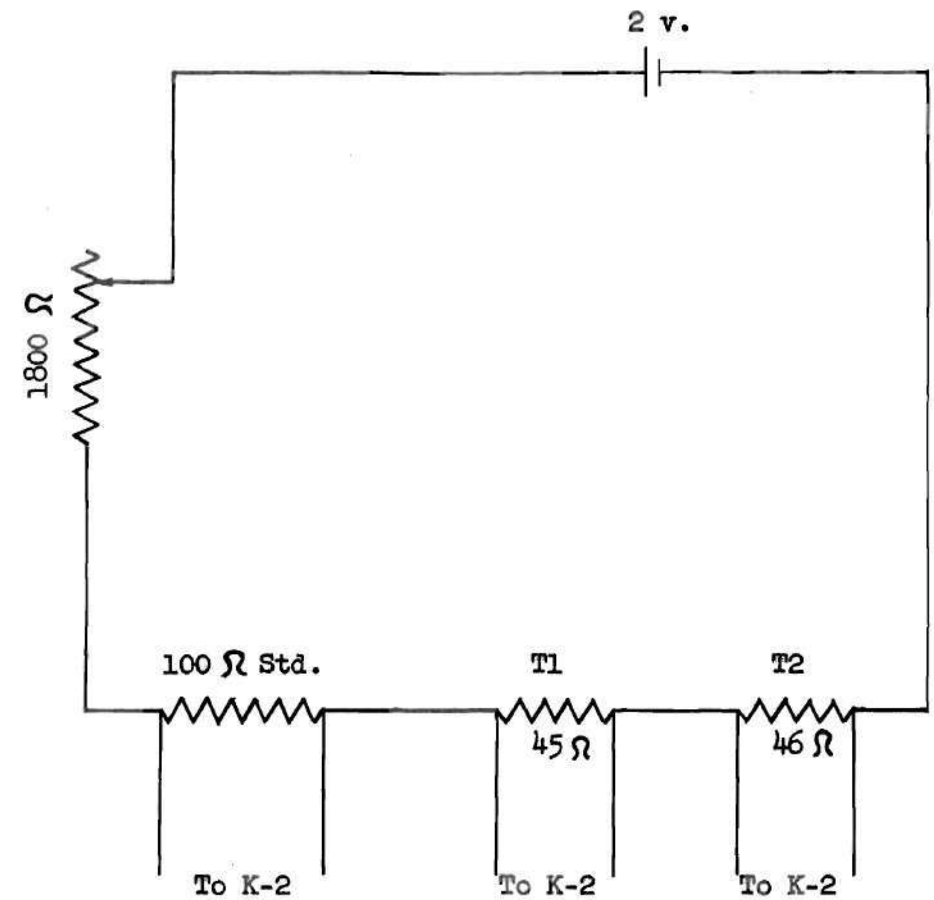

Figure 5

Carbon Resistance Thermometer Circuit

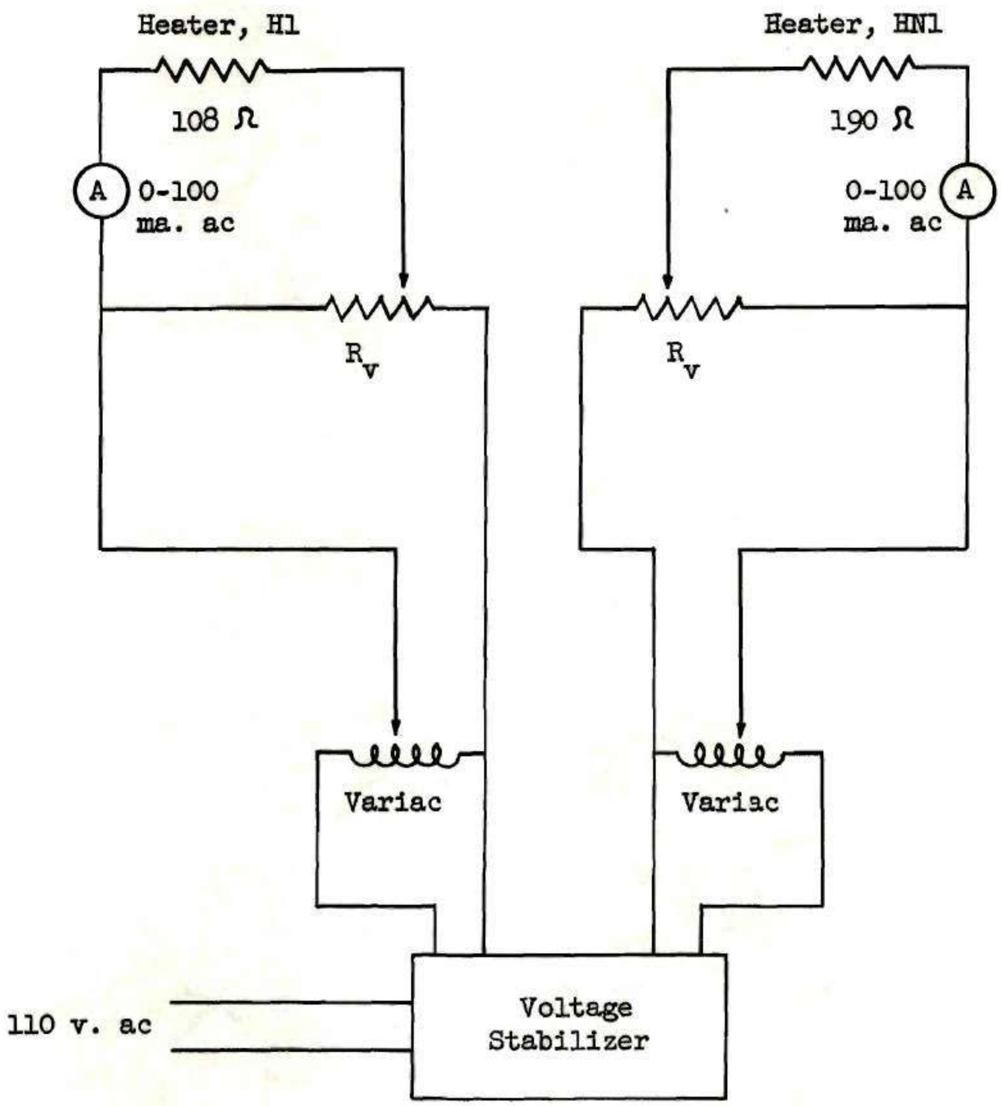

Figure 6

Secondary Heater Circuits

(Figure 6) shows an electrical circuit for only HN1 and H1, but the circuit for all three heaters is identical.

The heaters operated on 60 cycle alternating current available at 110 volts. The voltage was reduced by a variable auto-transformer (variac) and stabilized by a voltage stabilizer. Further control of the voltage and current was obtained by connecting the heaters in parallel with variable resistances (R_v). These resistances were Biddle rheostats. The variable resistances in parallel with the heaters could be used to vary the voltage across the heaters, and thus, permit better control of the current through the heaters than could be obtained with the variac alone.

CHAPTER IV

CALIBRATION OF THERMOMETRIC ELEMENTS

The thermometric elements of the cryostat were calibrated against a platinum resistance thermometer, which was obtained from the Low Temperature Laboratory of the Georgia Institute of Technology Engineering Experiment Station. The thermometric elements calibrated included two copper-constantan thermocouples (TC3 and TC4) and two carbon resistance thermometers (T1 and T2). The calibration experiments were made in the thermal conductivity cryostat, and data were taken in the temperature range 55° to 150°K.

In this temperature range, there was one data point which was easily reproduced. This point was the normal boiling point of liquid nitrogen (referred to hereafter as the fixed point), which was the refrigerant used to cool the cryostat. The remaining data points were taken at approximately ten degree intervals above and below this fixed point.

The e.m.f.-temperature data obtained were fitted to an equation given by Dike (12) in the form

$$E = at + bt^2 + ct^3 \qquad (4)$$

where E is the e.m.f. of the thermocouple, t is the temperature in °C, and a, b, and c are constants. Three of the data points were used in calculating the constants for the equation, and the remaining points were used as check points. Using this equation a temperature-e.m.f. table was constructed and used to convert the measured e.m.f. readings of the thermocouples to temperatures.

Platinum resistance thermometer.--The platinum resistance thermometer against which the thermometric elements were calibrated was previously calibrated in the Low Temperature Laboratory of the Georgia Institute of Technology Engineering Experiment Station and was labelled by this laboratory as "Th2". The calibration of the thermometer between 20° and 273°K was reported to agree to 0.01 degree with the National Bureau of Standards temperature scale. The calibration of the thermometer is reported in the Research Notes of Project 116[1].

The resistance of the platinum thermometer was measured directly in absolute ohms with a Leeds and Northrup, Type G-2 Mueller Bridge (Serial No. 740545), which was calibrated by the National Bureau of Standards in May, 1949 and certified to be correct to 2 parts in 100,000. This bridge was used in combination with a Leeds and Northrup, high sensitivity galvanometer (Serial No. 740876) and three 2-volt low discharge storage batteries connected in series. The maximum sensitivity obtained with the galvanometer and batteries was about 3.5 millimeters scale deflection per 0.0001 ohm, equivalent to approximately 0.001 degree.

This platinum resistance thermometer was previously used by Wright (4) as a primary standard for the calibration of a copper resistance thermometer and copper-constantan difference couples. Wright used

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[1]Research Notes of Project 116; Low Temperature Laboratory; Engineering Experiment Station; Georgia Institute of Technology; October, 1953 to February, 1955; pp. 15-16, 32, 69-75, 84.

a different apparatus for his calibration experiments. A brief comparison will be made between the results of his calibration of the copper-constantan difference couples and this calibration in a later section of this chapter.

<u>Cryostat assembly</u>.--For the calibration experiments the cryostat was identical with that shown in Figure 1 of Chapter II, except that no thermal conductivity specimen was attached to the copper plate EE. Also, the thermocouples and carbon resistance thermometers to be calibrated were attached to the platinum resistance thermometer well with two copper clamps designed especially for this purpose. To each of these clamps was attached one thermocouple and one carbon resistance thermometer. All metal-to-metal surfaces between the clamps and the thermometer well were coated with a thin layer of white petroleum jelly (vasoline) to insure good thermal contact between these surfaces. After the thermometric elements were in place, the monel shield (R2) and the copper shield (R1) were attached. The cryostat was then sealed by soldering the vacuum case (J) to the plate AA, and a large Dewar vessel to enclose the cryostat was mounted into position.

During the course of the calibration experiments, certain changes in the set-up of the cryostat were made in an attempt to improve the operating features of the cryostat and the reproducibility of the calibration data. The monel shield was removed after Run No. 3 to decrease the mass of the copper plate assembly. This change effectively reduced the time required to obtain a steady-state condition at each temperature point by about two hours. At the same time the inside of the copper shield was lined with a single layer of aluminum foil to decrease the transfer of heat by radiation between this shield and the copper plate assembly.

Another change in the cryostat was made after Run No. 5, because no success was obtained in reproducing the experimental values of the e.m.f. for the thermocouples and the resistance of the carbon resistance thermometers. This non-reproducibility of experimental data was attributed to two possible causes. They were (1) a partial electrical short circuit in the system and (2) a temperature gradient along the platinum resistance thermometer well at temperatures other than the fixed temperature of the refrigerant. Consequently, a test for short circuits was made on all electrical circuits. When this test proved negative, another copper clamp was attached to the thermometer well, and both thermocouples were attached to this clamp. The carbon resistance thermometers were not changed, because it was not practical to mount them on the same clamp. This change effectively improved the reproducibility of the thermocouple calibration data taken in the last two runs. The measured e.m.f. data at each temperature point were consistently the same over the entire temperature range. Also the difference between the e.m.f. values of the thermocouples at each temperature point was no longer erratic, but was constant to about 2.0 microvolts. Thus it was concluded that there did exist in previous runs a temperature gradient along the thermometer well at temperatures other than the fixed temperature of the refrigerant.

At the same time this change was made in the cryostat, a change was made in the external circuit for all thermocouple leads to the cryostat. The poles of the junction blocks were changed from brass to copper and wrapped with a loose cover of cotton to eliminate possible temperature effects at this junction.

A final change was made in the cryostat after Run No. 6 to determine the effects of the heat leak wire (F) on the operation of the system. The heat leak wire was disconnected from the copper plate, and at the same time another copper-constantan thermocouple TC5 was attached to the clamp to which the carbon thermometer T1 was attached. With the copper heat leak wire removed, the thermal connection between the plate and the pot consisted of the three monel support rods (N), which were spaced equidistant around the plate. This change effectively reduced the time required to adjust the system to steady-state conditions by about one hour for temperature points above the fixed point of the refrigerant. However, below the fixed point, the time was increased by about five hours at each point. The measured e.m.f. data for thermocouple TC5 at each temperature point were consistent with the e.m.f. data obtained for the other two thermocouples. The difference between the measured e.m.f. data for all three of the thermocouples also agreed to about 2.0 microvolts over the entire temperature range. Thus, since thermocouple TC5 was attached to a different clamp on the well, it was concluded that removing the heat leak wire eliminated the temperature gradient along the well. This set-up of the cryostat proved to give the best calibration data for both the thermocouples and the carbon resistance thermometers.

During the calibration experiments, the reference junctions of the thermocouples were maintained at a temperature of 0°C by use of an ice-water bath. This bath of commercial cracked ice and tap water was prepared in a pint Dewar flask and placed around the junctions. The junctions were then allowed to equilibrate in the bath for 30 to 45 minutes before the e.m.f. measurements of the thermocouples were started. At periodic intervals during the experiments water was drawn from the Dewar flask, and additional ice was added to insure that an equilibrium temperature was maintained.

The procedure used and the results of the calibration experiments can best be discussed by dividing the experiments into three temperature ranges. These ranges are: (1) at the normal boiling point of the refrigerant (referred to as the fixed point), (2) above the fixed point, and (3) below the fixed point.

At the fixed point.--After the cryostat was assembled and sealed, the cryostat was evacuated to about 1×10^{-6} mm Hg. The Dewar vessel around the cryostat was filled until the liquid nitrogen was one to four inches above the top of the vacuum case. It was necessary to keep this vessel filled to approximately this level throughout the experiments to prevent heat leaking down the tubes into the cryostat. Helium exchange gas was then admitted into the cryostat, thus breaking the high vacuum, but providing a means of cooling the cryostat rapidly. When the cooling rate of the cryostat began to decrease, the refrigerant pot P was filled with liquid nitrogen, and the system was allowed to continue cooling. During this cooling period, which lasted approximately one hour, the temperature difference between the copper plate and the refrigerant pot was followed by the thermocouple TC3 and the difference couple TD1. At the end of this cooling period as noted by a temperature difference between the copper plate and the refrigerant pot of 0.5 to 1.5 degrees (7 to 20 microvolts), the helium exchange gas was pumped from the system, and the system was evacuated again to 1×10^{-6} mm Hg or better. The system was allowed to equilibrate for about one hour. During this time the temperature of the copper plate was followed to obtain some idea of the drift rate. This was accomplished by measuring the e.m.f. of thermocouple TC3 or by measuring the resistance of the platinum resistance thermometer TS. When the drift rate was 0.01 degree per minute or less (Δe.m.f. = 0.2 microvolt/min. or ΔR = 0.0013 ohm/min.), a series of calibration data at the fixed point was taken.

A series of calibration data consisted of measuring the resistance of the platinum resistance thermometer and the e.m.f. across the 100-ohm standard resistance, the carbon thermometers T1 and T2, and the thermocouples TC3 and TC4 in the order named as a function of time. These measurements were repeated four times and ended by measuring the resistance of the platinum thermometer. These four e.m.f. measurements were then averaged or extrapolated to one temperature within the data to determine an average e.m.f. value at this temperature. A sample calculation of e.m.f. measurements to an average e.m.f. is given in Appendix B. The results of the calculation for each thermocouple are shown in Table 6. The corresponding data for the carbon resistance thermometers are given in Appendix D.

Table 6

Average E.M.F. of
Thermocouples at the Fixed Point

Run No.	Point No.	Temperature, °K	Average e.m.f., microvolts TC3	TC4	TC5
5	5-2	77.604	5093.9	5095.3	
6	6-1	77.663	5091.7	5093.0	
6	6-7	77.948	5090.2	5091.6	
4	4-1	77.990	5088.7	5090.1	
7	7-1	78.057	5087.2	5088.8	5087.6
7	7-6	78.105	5086.0	5087.5	5086.4
7	7-12	78.265	5084.9	5086.2	5085.2
5	5-8	78.362	5083.1	5084.0	
5	5-1	78.434	5082.6	5084.0	
2	2-1	78.471	5081.8	5082.9	
4	4-12	78.765	5077.8	5079.0	
5	5-10	79.089	5067.3	5070.5	
4	4-8	79.570	5065.5	5066.9	

Using the data given above a plot of e.m.f. versus temperature showed that these data were fitted by a straight line. This was expected since the sensitivity of a thermocouple is essentially constant over a small temperature difference. All the data fit this line to within $\pm$ 1.0 microvolt except data of Point No. 5-10, which deviated about -5.0 microvolts. Since no explanation was apparent for the deviation of this data point, it was attributed to a non-recurring error in measurements and excluded from future considerations of the data.

From this plot of e.m.f. versus temperature, the sensitivity of the thermocouples was determined to be 14.8 microvolts per degree. Also, by extrapolating and averaging the data at a temperature of 78.265°K the e.m.f. of thermocouples TC3, TC4, and TC5 were found to be 5084.6, 5085.8, and 5084.6 microvolts, respectively. The standard deviation from these average e.m.f. values, i.e. the limits within which two-thirds of the data fell, was calculated to be $\pm$ 0.7 microvolt for thermocouples TC3 and TC4. Since there were only three calibration points at this temperature for thermocouple TC5, this deviation was also used for it. Thus the calibration data at the fixed point was reproducible to $\pm$ 0.7 microvolt, which corresponded to $\pm$ 0.05 degree in the temperature.

Above the fixed point.--The calibration points above the fixed point were complicated by the fact that the copper plate was no longer at the temperature of the refrigerant pot. The temperature of the plate was raised by a heater at about ten degree intervals up to approximately 150°K. Thus there was a flow of heat from the copper plate to the refrigerant pot. It was the control and balance of this heat flow which complicated these measurements.

From the fixed point the temperature of the plate was raised about ten degrees (about 200 microvolts) by supplying power to the copper plate heater H1. During the initial heating period maximum current (100 milliamperes) was allowed to flow through the heater, and the temperature of the plate was followed by reading the e.m.f. of thermocouple TC3. The initial heating rate of the plate varied from eight to ten microvolts per minute and depended on the magnitude of the temperature difference between the plate and the pot. The rate decreased as the temperature difference increased. At the end of this heating period (20 to 25 minutes), the current through the heater was reduced, thus decreasing the power to the heater and the heat flow to the plate. Continued adjustment of the current was made until the heat flow to the plate was balanced with the heat flow from the plate. To determine the balance of these heat flows, the drift rate of the temperature difference between the plate and the pot was followed with the difference couple TD1 and the thermocouple TC3. This drift was a change in the temperature of the copper plate, since the temperature of the pot was essentially held constant by the refrigerant. When the drift rate was $\pm$ 0.01 degree per minute ($\pm$ 0.2 microvolt per minute), the heat flows were considered balanced, and the system was allowed to equilibrate for 30 to 45 minutes to insure that the system was in a steady-state condition. After this equilibrium period calibration data were taken at this temperature point as previously discussed. This procedure was then repeated at approximately ten degree intervals up to about 150°K, until data were obtained at seven or eight temperature points in the range 78° to 150°K.

After obtaining the data above the fixed point, the system was cooled to the fixed point with helium exchange gas or was allowed to cool by conduction along the heat leak between the plate and the pot. Usually helium exchange gas was used, because the cooling was more rapid. When the fixed point was reached and the system was again at equilibrium conditions, a series of calibration data was usually taken. Table 7 is a tabulation of the average e.m.f. data taken at temperatures above the fixed point. These data were calculated as described in the sample calculation of Appendix B.

Table 7

Average E.M.F. of
Thermocouples Above the Fixed Point

Run No.	Point No.	Temperature, °K	Average e.m.f., microvolts TC3	TC4	TC5
7	7-7	87.295	4944.1	4945.1	4944.1
6	6-2	98.819	4755.0	4755.8	
7	7-2	101.022	4715.8	4715.2	4714.6
7	7-8	112.171	4510.5	4509.8	4509.1
7	7-9	121.525	4328.6	4327.1	4326.4
7	7-3	130.416	4146.3	4145.0	4143.7
6	6-3	131.510	4124.1	4125.3	
7	7-4	143.472	3860.2	3858.2	3856.2
6	6-4	146.455	3792.5	3793.6	
7	7-5	151.534	3673.1	3670.9	3668.9

Included in this table are data for Runs No. 6 and 7 only. Data taken prior to these runs were not included, because they were not used in the determination of the final e.m.f.-temperature relationship. Reasons for discarding these data were previously discussed in this chapter.

The sensitivity of the thermocouples was determined at data points where the temperature difference between the points was small enough to assume the sensitivity was essentially constant. For example, using the experimental data of Point Nos. 6-2 and 7-2, which was calculated to give the average e.m.f. data of Table 7, the sensitivity at an average temperature of 99.4°K was 16.8 microvolts per degree. In this way the sensitivity of the thermocouples at temperatures above the fixed point was calculated and found to increase from 14.8 microvolts per degree at 78.3°K to 23.1 microvolts per degree at an average temperature of 145.0°K.

The reproducibility of the e.m.f. data at each temperature point above the fixed point could not be evaluated with any degree of confidence, because of the few measurements made at each point. Since there was no known reason to believe that the data above the fixed point were any less reproducible than at the fixed point, it was assumed that these data were also reproducible to $\pm$ 0.05 degree.

Below the fixed point.--The temperature points below the fixed point were obtained by reducing the pressure above the refrigerant in the pot, thus reducing the temperature of the refrigerant and the pot. The copper plate was cooled by conduction, since the plate was connected thermally to the pot. Calibration data below the fixed point were obtained in this manner at approximately eight degree intervals in the temperature range of 55° to 78°K.

The refrigerant pot was cooled to temperatures below the fixed point by reducing the pressure over the refrigerant with a mechanical vacuum pump. The pot was filled with refrigerant to about three-quarters full and slowly evacuated to about 28.0 inches of mercury. Pumping on the pot was continued at this rate until all the data in this temperature range were obtained. The copper plate was cooled by conduction along the heat leak wire and monel support rods between the plate and the pot. When the plate was cooled about eight degrees, the plate heater H1 was turned on. The system was adjusted to steady-state conditions as discussed in the previous section, and calibration data were taken at this point. The heater was then turned off and the plate was allowed to cool about eight more degrees. At this point the system was again adjusted to steady-state conditions by energizing the plate heater and calibration data were obtained. After obtaining data at this point, the heater was turned off and the system was allowed to continue cooling until the temperature of the plate was drifting about 0.01 degree per minute or less. Calibration data were then taken at this temperature point, which was the lowest attainable point below the fixed point.

After obtaining calibration data at the lowest point, the refrigerant pot, while still under a vacuum, was heated to the fixed point with the heater HN1. At this temperature the vacuum in the pot was zero inches of mercury, when there was liquid nitrogen remaining in the pot. The pot was then opened to the atmosphere through a calcium chloride drying tube. The system was warmed to the fixed point with exchange gas and allowed to equilibrate. A series of calibration data was then taken at the fixed point. This was the last calibration point for each run. The cryostat was then allowed to warm to room temperature and opened to the atmosphere.

In Table 8, the average e.m.f. of the data taken at temperature points below the fixed point is shown. The calculation of these data is discussed in Appendix B.

Table 8

Average E.M.F. of
Thermocouples Below the Fixed Point

Run No.	Point No.	Temperature, °K	Average e.m.f., microvolts TC3	TC4	TC5
6	6-6	55.880	5384.2	5386.3	
7	7-11	63.436	5293.0	5294.6	5293.6
6	6-5	67.019	5244.7	5246.8	
7	7-10	71.827	5181.0	5181.8	5184.4

The data shown in this table are for Runs No. 6 and 7 only, because data taken prior to these runs were not used to determine the final e.m.f.-temperature expression. Reasons for discarding these data were previously discussed in this chapter.

The sensitivity of the thermocouples was evaluated in the same manner as discussed for data taken above the fixed point. It was found that the sensitivity was 13.4 microvolts per degree at an average temperature of 65.2°K.

It was also not possible to evaluate the reproducibility of the data below the fixed point with any degree of confidence, because of the few measurements made at each point. Thus, it was assumed that the data were reproducible to $\pm$ 0.05 degree.

Accuracy of calibration data.--The accuracy of the calibration data for the thermocouples was effected by the reproducibility of the e.m.f. of the thermocouple, the precision of the potential measurements, the accuracy of the temperature measurements, and the precision of the measuring instruments.

The precision of the potential measurements for each thermocouple was dependent on the sensitivity of the galvanometer system used with the potentiometer and the reading of the scale of the potentiometer. The sensitivity of the galvanometer system was about 0.6 microvolt per millimeter scale deflection over the entire temperature range; thus, a change of $\pm$ 0.3 microvolt in the e.m.f. should be easily determined. Since the scale of the potentiometer could be read to $\pm$ 0.1 microvolt, the precision of the potential measurements was taken as $\pm$ 0.3 microvolt.

The accuracy of the temperature measurement was dependent primarily on the effects of temperature gradient along the copper well which surrounded the resistance thermometer and to which the thermocouples were attached. No direct method was available for determination of this effect. It was estimated that these effects did not introduce an error of ± 0.01 degree over the entire temperature range of the calibration. In view of the high thermal conductivity of copper and the low heat flows in the copper plate assembly this would seem a conservative estimate for the effect of temperature gradient under steady-state conditions. This error of ± 0.01 degree was equivalent to ± 0.1 microvolt at the lowest temperature and ± 0.2 microvolt at the highest temperature.

The error introduced as a result of the accuracy of the potentiometer was fixed by the certification accompanying this instrument, which was ± 0.02 per cent. This was equivalent to ± 1.1 microvolt at the lowest temperatures and ± 0.7 microvolt at the highest temperatures.

As previously discussed, the e.m.f. of the thermocouples was reproducible to ± 0.05 degree or ± 0.6 microvolt at the lowest temperature and ± 1.2 microvolts at the highest temperatures. Thus the overall accuracy of the calibration data was ± 2.1 microvolts at the lowest temperatures and ± 2.4 microvolts at the highest temperatures. In terms of temperature this is equivalent to ± 0.17 degree at 55°K and ± 0.10 degree at 150°K.

<u>E.M.F.-temperature relation</u>.--The e.m.f.-temperature relation was first derived after obtaining the data of Runs No. 2, 3, and 4. The constants in equation (4) were evaluated by using three of the experimental data

points of these runs. When the experimental data of Runs No. 6 and 7 were found to deviate from this equation by about 30 microvolts at the highest temperatures, a correction was applied to the equation to make it fit the data of Runs No. 6 and 7. This correction was based on a plot of the difference between the calculated and experimental e.m.f. and the temperature. The resulting corrected equation was then converted from degrees Centigrade to degrees Kelvin[2]. The final equation is

$$E = 5879.7 - 5.3973\ T - 0.060976\ T^2 + 3.02 \times 10^{-6}\ T^3 \qquad (5)$$

where E is the e.m.f. in absolute microvolts and T is the temperature in degrees Kelvin. The coefficients of the second and third terms contain one more significant figure than the data warrant but are carried to permit calculation to 0.1 microvolt.

This equation was used to calculate the e.m.f. corresponding to the temperatures of the data points shown in the preceding tables of this chapter. The results indicate that the equation fits the experimental data of all three thermocouples to within 0.08 per cent over the temperature range of the calibration. Further, a plot of experimental temperature minus calculated temperature versus temperature (see Figure 7) indicated the equation may be expected to give temperatures to within $\pm$ 0.15 degree in the range 60° to 150°K.

The slope of equation (5),

$$\frac{dE}{dT} = -5.40 - 0.1220\ T + 9.06 \times 10^{-6}\ T^2 \qquad (6)$$

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[2]The ice point was taken to be 273.16°K.

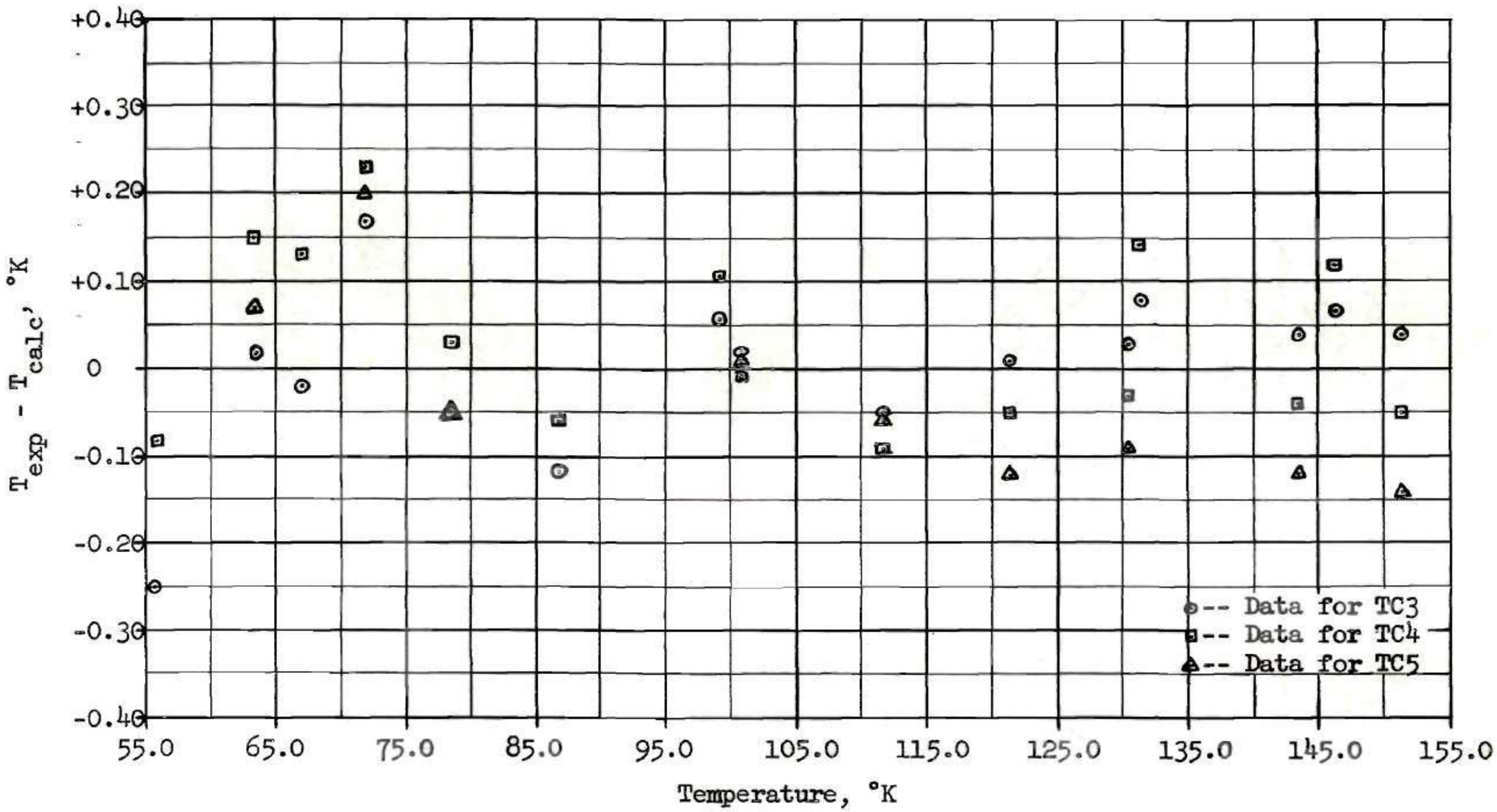

Figure 7

Deviation of Experimental Temperature of Thermocouples
from
Calculated Temperature (Eq. 5)

indicated that a temperature difference of one degree would change the e.m.f. calculated in the first decimal. Therefore, it was necessary in constructing an e.m.f.-temperature table to tabulate values for each degree in order to obtain an accuracy of 0.1 microvolts. Values falling between each degree may then be calculated by linear interpolation of the table values. The e.m.f.-temperature table is given in Appendix A as Table 10.

Comparison to Wright's calibration of copper-constantan difference couples.--Wright (4) in his work used two three-junction, copper-constantan difference couples for measurement of temperature in the range 78° to 300°K. These couples were constructed from wire taken from the same spools as was used in the present research. He calibrated these couples against the same platinum resistance thermometer as used in this work, but in a different apparatus and using a technique which permitted only differences in temperature to be measured with the thermocouple. Since the thermoelectric power of a thermocouple is directly proportional to the number of junctions, the results of Wright's calibration were divided by three and are shown in Figure 8. This figure shows that the sensitivity, i.e. dE/dT found by Wright for his thermocouples was 1.0 to 2.0 microvolts less than that obtained in this study. The reason for this discrepancy is not known, but may lie in the different techniques used to calibrate the couples or perhaps in the fact that strains were introduced by Wright in making his three-junction difference couples. Further experiments are planned to examine these points by an actual comparison of Wright's couples in the present cryostat.

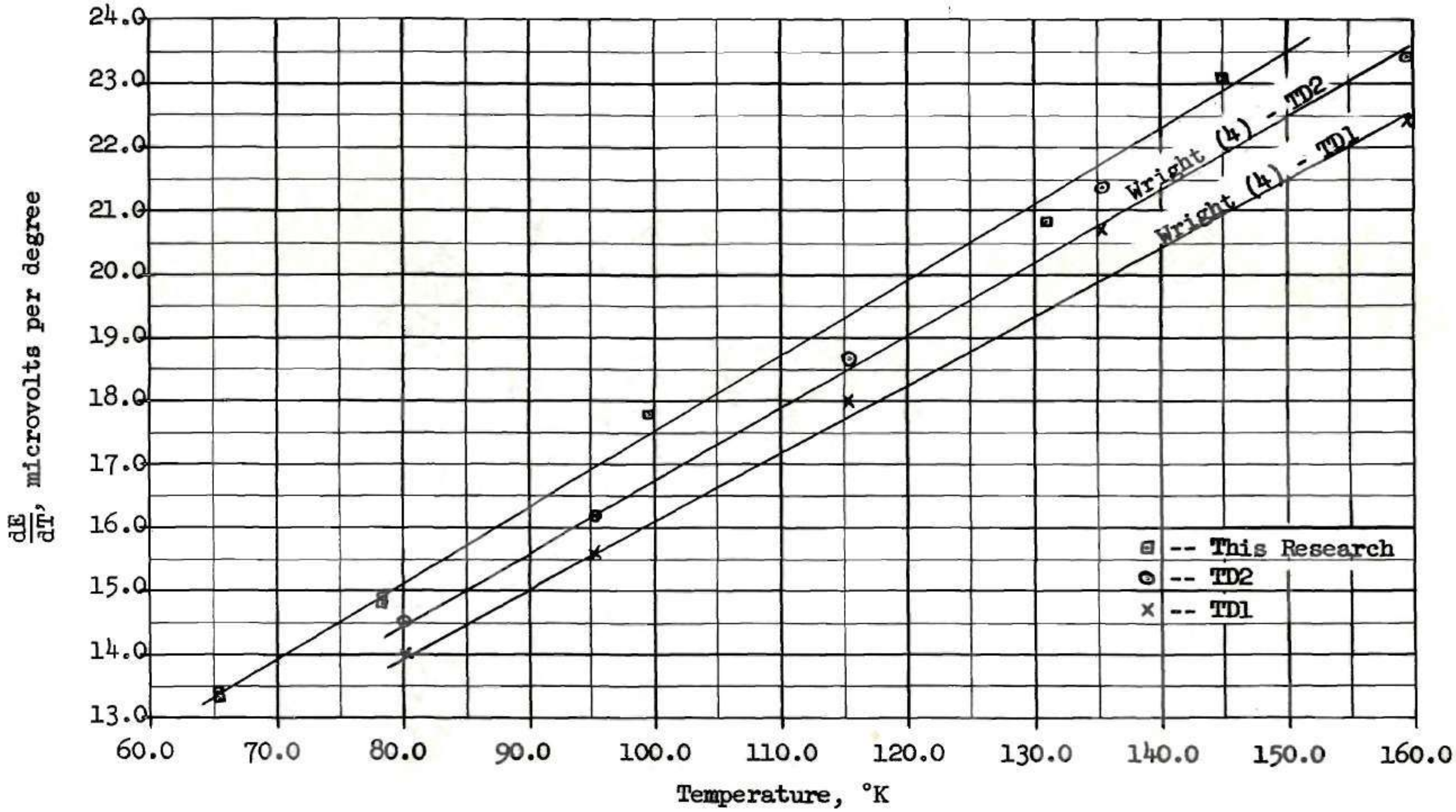

Figure 8

Thermoelectric Power of Thermocouples

CHAPTER V

THERMAL CONDUCTIVITY MEASUREMENTS AND RESULTS

A measurement of the thermal conductivity of a metal consists of measuring under steady-state conditions the temperature difference between two fixed points on a specimen which is established by a measured flow of heat through the specimen. The ratio of the temperature difference to the distance between the two fixed points is the temperature gradient along the specimen and under steady-state conditions is constant. The thermal conductivity at the mean temperature of the specimen is the ratio of the flow of heat through the specimen to the temperature gradient along the specimen and to the area of the specimen normal to the flow of heat. This assumes that the thermal conductivity is linear in temperature over the temperature difference used. In practice, these temperature differences are usually made small enough so that this assumption is valid. This procedure is very simple in principle, but experimentally is complicated by the determination of such factors as heat losses due to conduction along the leads of the heater and thermometers and to radiation to surrounding bodies.

Thermal conductivity specimen.--The specimen used in these experiments was a commercial free-machining yellow brass rod, 1/4 inch in diameter and 7 7/8 inches long. This specimen was made from the same rod from which Wright (4) made his yellow brass specimen and was used so that a comparison could be made with the thermal conductivity as determined by Wright. This was thought to be a convenient method of checking the data obtained with this cryostat and at the same time a check on the apparatus used by Wright.

The nominal composition of the specimen was 62 per cent copper, 35 per cent zinc, and 3 per cent lead. It was made from a drawn brass rod taken from stock which was of half-hard temper. Alloys of this composition fall in the α-brass region at room temperatures on a phase diagram of copper-zinc alloys.

Cryostat.--The cryostat was set up as shown in Figure 1, Chapter II except that the monel radiation shield R2 was removed, since experience gained during the thermocouple calibration studies had shown that the presence of this shield gave a very slow approach to steady-state conditions. The inside of the copper radiation R1 was wrapped with a loosely fitting layer of aluminum foil. The specimen rod was soldered with Wood's metal to a copper disc which was attached to the copper plate with screws. The metal surface of the disc in contact with the plate was coated with white petroleum jelly (vasoline) to insure good thermal contact between these surfaces. Small copper clamps to which the copper-constantan thermocouples TC3 and TC4 were attached were fitted about five inches apart on the specimen. These metal-to-metal surfaces were also coated with white petroleum jelly. The heater was soldered to the free end of the specimen with Wood's metal and was then wrapped with a loose cover of aluminum foil to decrease loss of energy by radiation. This assembly was then enclosed in the vacuum jacket, and the entire cryostat assembly immersed in a large Dewar vessel containing liquid nitrogen.

<u>Procedure</u>.--The procedure for operating the cryostat was similar to the discussion given in the chapter on calibration of the thermometric elements, but differed from it by the addition of the control of another heater, i.e. the specimen heater HT. The control of this heater proved to be relatively simple after experience gained in an initial experimental run.

The first step in this procedure after assembling the cryostat was to evacuate the cryostat to a high vacuum (1×10^{-6} mm Hg) and then to cool the cryostat with the liquid refrigerant. During the initial cooling helium exchange gas was admitted to the cryostat to provide a rapid means of cooling. This gas was evacuated after the cryostat was cooled to the temperature of the refrigerant. Liquid refrigerant was also added to the refrigerant pot inside of the cryostat and was present in the pot during all experimental runs. This pot was the heat receiver for the heat from the copper plate and specimen.

After cooling and evacuating the cryostat, the next step was to establish a temperature gradient along the specimen. This was done by energizing the specimen heater. The current through this heater was set at about 21 milliamperes, and the system was allowed to stand until steady-state conditions were attained in the heat flow through the specimen. During this time the temperature of the specimen was followed by reading the e.m.f. of thermocouples TC3 and TC4 and the difference couple TD1. After approximately four hours, the temperature of each thermocouple was changing at a rate of 2.0 microvolts per hour or about 0.12 degree per hour. At this time the temperature difference between the two thermocouples was about 90 microvolts or 5 degrees, and the mean temperature was approximately 89°K. This very small drift rate in the temperature was considered to be small enough to assume that the system was at steady-state conditions. A series of measurements was then taken. These measurements included the e.m.f. across the specimen heater and the 1-ohm standard resistance in series with the heater for calculation of the power input to the heater and the e.m.f. of the thermocouples TC3 and TC4 for the determination of the mean temperature and the temperature difference at two points along the specimen. A sample sheet of data taken during steady-state conditions is shown in Table 15, Appendix C.

After obtaining data at this temperature point, the temperature of the copper plate was raised about ten degrees (200 microvolts) by the copper plate heater. This, in turn, raised the mean temperature of the specimen about ten degrees, and thus it was possible to obtain experimental data at ten degree intervals up to approximately 150°K. The specimen heater was already adjusted to establish a temperature difference of approximately five degrees between the two thermocouples, thus it was only necessary to regulate the copper plate heater and then to allow the system to stand until steady-state conditions were attained, before experimental data were taken at each temperature point.

Using this procedure, experimental data were obtained at four temperature points at mean temperatures of 89° to 118°K. During these four runs the power input to the specimen heater was constant at approximately 0.069 watts, and the temperature difference between the two thermocouples was about five degrees. Approximately six hours were required to obtain data at each temperature point, but it is felt that this time could have been shortened considerably with more experience in the operation and control of the heaters. The drift rate at steady-state conditions for each of these runs was not greater than 0.12 degree per hour except for Run No. 2 when it was approximately 0.36 degree per hour. The drift rate was warming in Runs No. 1, 2, and 3 and cooling in Run No. 4.

After obtaining data at these four points, the heaters were switched off and the cryostat was cooled again to the temperature of the refrigerant. The specimen heater was switched on, and the current adjusted so that about 15 milliamperes passed through the heater. The system was then allowed to stand for several hours until steady-state conditions were attained after which experimental data was taken. The drift rate for this run (Run No. 5) was 1.7 microvolts per hour (0.11 degree), and the system was cooling. The temperature difference between the two thermocouples was about three degrees, and the mean temperature was approximately 85°K.

<u>Results</u>.--The thermal conductivity of yellow brass as determined in this work is presented in Table 9. ΔT is the difference between two fixed points in the heat flow path; T_m is the mean temperature of the temperatures at the two fixed points; and k_m is the mean thermal conductivity of the brass specimen at T_m. A sample calculation is given in Appendix C, which illustrates the method of obtaining the data in Table 9 from the experimental data.

Table 9

Mean Thermal Conductivity of Yellow Brass

Run No.	T_m, °K	ΔT, °K	k_m, watts/cm-°K
5	84.67	2.93	0.486
1	88.73	5.52	0.498
2	98.90	5.11	0.528
3	109.01	4.91	0.564
4	118.24	4.64	0.605

The data of the above table are plotted in Figure 9 and labelled as (A). Examination of this figure shows that a straight line could be drawn through these data. Further, the data fitted this line to within two per cent, which is believed to be well within the overall accuracy of the conductivity data.

Wright (4) has also made thermal conductivity measurements on a yellow brass specimen cut from the same rod as that used in these experiments. The results of his measurements are also shown in Figure 9. His measurements were made in a cryostat of different design, which operated in principle the same as this cryostat. His specimen was shorter (about 2 1/2 inches long), and he used temperature differences of approximately the same magnitude as Run No. 5 in these experiments. Comparison of the results obtained by these two methods shows that Wright's thermal conductivity data are lower by approximately ten per cent over the range of these experiments. The slope of the two lines are in general the same suggesting that the deviation between these two sets of measurements may be due to a systematic error. This discrepancy is discussed in a later section.

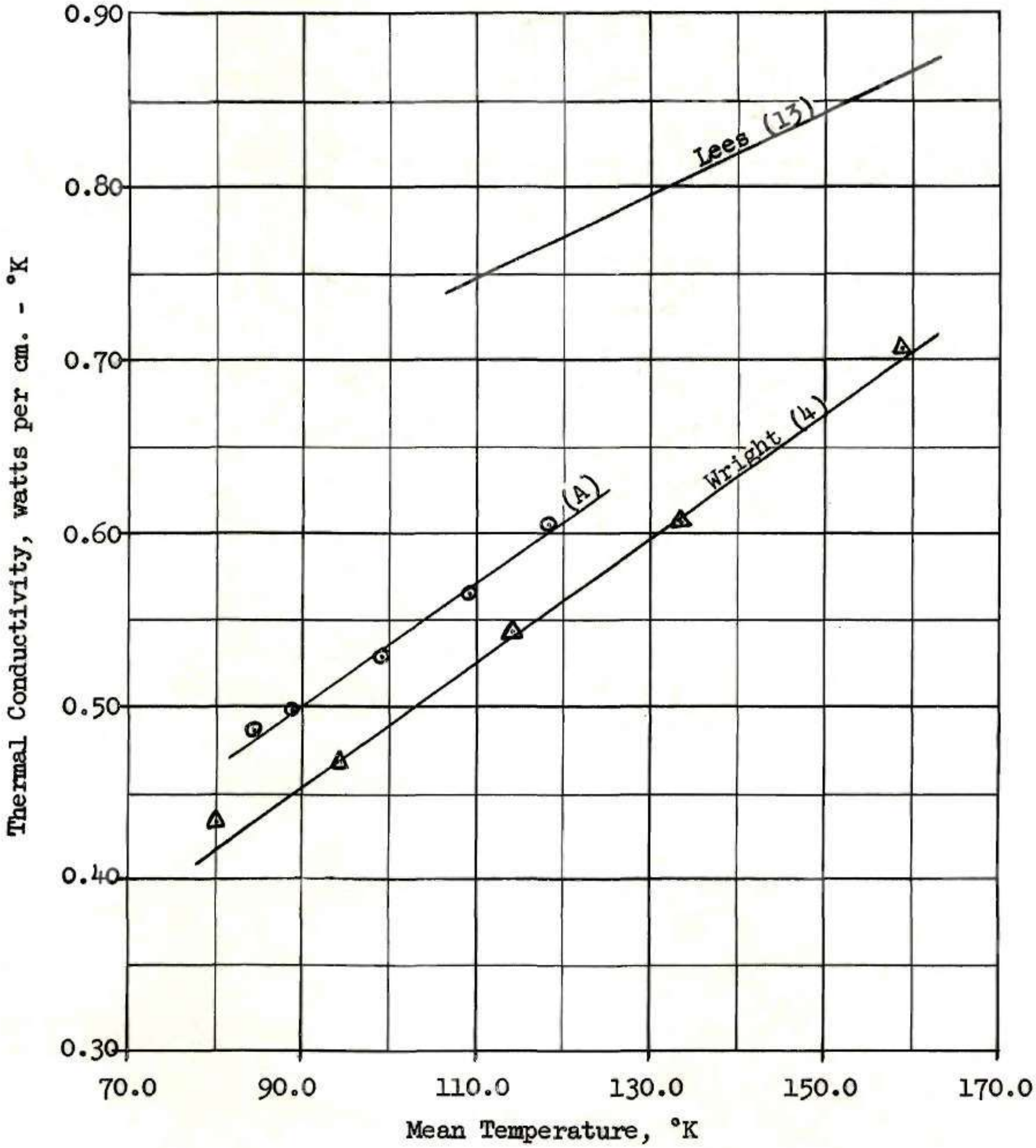

Figure 9

Thermal Conductivity of Yellow Brass

Thermal conductivity data of Lees (13) in Figure 9 are shown for a brass of composition 70 per cent copper and 30 per cent zinc. This represents the only data reported in the literature for yellow brass over this temperature range. There are, however, scattered data reported for temperature points such as room temperature and boiling point of liquid nitrogen. No data were available on a leaded brass, such as used in this work.[1]

Accuracy of results.--The primary sources of error in the thermal conductivities given in Table 9 were due to the uncertainty in the measurement of the temperature gradient along the specimen and the uncertainty in the radiation losses. The total error introduced into the thermal conductivity as a result of error in the heat input to the specimen heater, cross-sectional area, and length of the specimen was less than 0.5 per cent.

The temperature difference was obtained as the difference in temperature of two thermocouples each of which may have been in error by as much as 0.15 degree. Since the temperature differences measured were approximately five degrees, random errors of $\pm$ 0.15 degree could have introduced an uncertainty of as much as 0.3 degree or six per cent in

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[1]Since this work was completed measurements of the thermal conductivity of a leaded brass (61.0 per cent copper, 35.7 per cent zinc, and 3.3 per cent lead) of hard-temper have been reported by Powell, Robert L., H. Martin Roder, William M. Rodgers, "Low-Temperature Thermal Conductivity of Some Commercial Coppers," Journal of Applied Physics, 28, (1957), p. 1282-1288. These measurements, which extended from 4° to 120°K, are about 12 per cent lower than those found in the present work. The lower values may be due to differences in composition or the state of strain.

the mean thermal conductivity. Actually the smoothness of the data shown in Figure 9 (Curve A) indicates that random errors of this magnitude were not present.

The error due to radiation losses (which were not corrected for) is more difficult to assess. At the lowest temperatures (approximately 80°K) the radiation loss is certainly negligible. At the highest temperature radiation losses from the specimen and heater are probably present.

It is possible to explain part of the disagreement between the present measurements and those of Wright in terms of the difference of sensitivities of the thermocouples as shown in Figure 8, Chapter IV. Since these thermocouples were all constructed from the same wires, one might reasonably expect the thermocouples to exhibit the same sensitivity. If one assumes that the sensitivity of Wright's couples are in fact the same as those used in the present work, then the values of thermal conductivity computed by Wright should be increased by about six per cent. Such an increase would bring the two sets of measurements into an agreement well within the expected remaining errors of either set. It is suggested that a direct comparison of the sensitivity of the thermocouples used in this thesis and those used by Wright be made to settle this point.

APPENDIX A

TABLES

Table 1

Dimensions and Weight of Cryostat Parts

Name of Part	Symbol in Fig. 1	Dimensions O.D., Inches	Dimensions Wall Thickness, Inches	Dimensions Depth, Inches	Weight,[a] Grams
Top of Vacuum Case	AA	4	--	3/32	--
Vacuum Case	J	4	0.065	20 1/8	--
Top of Charcoal Can	BB	2	--	1/16	--
Top of Refrigerant Pot	CC	3	--	3/32	--
Charcoal Can	K	2	0.030	3 3/4	133[b]
Refrigerant Pot	P	3	0.030	2 1/2	151[c]
Bottom of Refrigerant Pot and Charcoal Can	DD	3 1/2	--	3/32	235[d]
Calorimeter Ring	L	3	0.065	3/4	(d)
Copper Plate	EE	3	--	1/8	179[e]
Copper Plate Heater Ring	H1	3	0.035	13/16	47
Platinum Thermometer Well	TS	5/8	0.125	2 3/4	(e)
Specimen Heater Shell for,					
1/4 in. dia. specimen	HT	7/8	--	7/8	37
3/8 in. dia. specimen	HT	7/8	--	7/8	36
Monel Radiation Shield	R2	3	0.035	8 1/2	475
Copper Radiation Shield	R1	3 1/2	0.011	12	214
Specimen	S	1/4 or 3/8	--	Up to 7 7/8	--
Platinum Thermometer	TS	--	--	--	20

(a) These weights apply to the parts before final assembly.

(b) Weight of charcoal can includes top (BB), but does not include bottom of can (DD).

(c) Weight of refrigerant pot includes top (CC), but does not include bottom of pot (DD).

(d) Weight of bottom of refrigerant pot and charcoal can (DD) and calorimeter ring (L).

(e) Weight of copper plate (EE) and platinum thermometer well (TS).

Table 2

Tubes in Cryostat

Tube[a] Diameter, Inch	Kind of Tube	Symbol in Fig. 1	Use
1/2	Monel	V1	Thermal conductivity cryostat wire seal and vacuum line
1/2	Monel	Z	Gas thermometer capillary exit line
1/4	Monel	W1	Thermal conductivity cryostat wire seal line
1/4	Monel	V2	Calorimeter vacuum line
1/4	Monel	X	Extra line
3/16	Monel	V3	Refrigerant pot filling and vacuum line
3/16	Monel	V4	Charcoal can filling and vacuum line
3/16	Monel	U	Refrigerant pot vent line
1/8	Super Nickel	W2	Calorimeter wire seal line

(a) All tubing had 0.010 inch wall thickness.

Table 3

Electrical Leads into Cryostat

Description of Wires	No. of Wires	Use
B&S No. 34 gauge, single cotton, enameled copper	4	Platinum thermometer
	8	Two carbon resistance thermometers
	4	Specimen heater
	2	Copper plate heater
	2	Monel radiation shield heater
	4	Two difference couples
	5	Five thermocouples
	7	Spares
B&S No. 30 gauge, single cotton, enameled copper	2	Refrigerant pot heater
	2	Spares
B&S No. 32 gauge, double nylon, enameled constantan	5	Five thermocouples
	5	Spares

Table 4

Description of Heaters in Cryostat

	Symbol in Fig. 1	Max. Current, ma.	Size of[1] Wire B&S No.	Resistance, Ohms	Turns
Specimen Heater,					
For 1/4 in. dia. Specimen	HT	100	36	160	56
For 3/8 in. dia. Specimen		100	36	150	55
Copper Plate Control Heater	H1	100	32	108	30
Monel Radiation Shield Heater	H2	100	30	119	50
Refrigerant Pot Heater	HN1	200	30	190	80

(1) The wire used for making all heaters is double nylon, enameled constantan wire.

Table 5

Instruments

Item	Manufacturer	Serial Number	Calibration
Type K-2 Potentiometer	Leeds & Northrup	679633	Not calibrated in an absolute sense, but certified to be accurate to 0.02 per cent.
High Sensitivity Galvanometer	Leeds & Northrup	643992	Sensitivity, 0.32 microvolts per mm. Critical damping resistance (C.D.R.X.) 49 ohms Period, 6.0 sec. resistance, 12 ohms.
Galvanometer	Leeds & Northrup	--	Pointer type, horizontal scale Sensitivity, 3.2 μv/mm. Critical damping resistance (C.D.R.X.), 50 ohms Period, 2.4 sec. Resistance, 20 ohms
Volt Box	Leeds & Northrup	777854	Correct to within 0.01 per cent, April, 1950.
Standard Resistor	Leeds & Northrup	649498	1.00044 absolute ohms, November, 1952.
Resistance Box	Leeds & Northrup	713057	0-9999 ohms.
Standard Resistor	Rubicon	50037	100.002 abs. ohms. June, 1949.
Resistance Box	Rubicon	39506	
Standard Cell	Eppley Laboratories	323984	1.01879 international volts at 25°C December, 1945.
Rheostats	Biddle	92740	896 ohms, 0.7 amps.
		95426	857 ohms, 0.7 amps.

Table 10

Temperature-EMF Relation of Copper-Constantan Thermocouples

$$E = 5879.7 - 5.3973\ T - 0.060976\ T^2 + 3.02 \times 10^{-6}\ T^3$$

Reference Junction = 0°C   E in absolute volts
Ice Point = 273.16°K $1\ \mu v. = 10^{-6}$ volts

T, °K	E, μv.	1st Diff.	T, °K	E, μv.	1st Diff.	T, °K	E, μv.	1st Diff.
55.00	5398.9	12.1	88.00	4934.7	16.1	121.00	4339.4	20.1
56.00	5386.8	12.3	89.00	4918.6	16.2	122.0	4319.3	20.2
57.00	5374.5	12.4	90.00	4902.4	16.4	123.00	4299.1	20.3
58.00	5362.1	12.5	91.00	4886.0	16.5	124.00	4278.8	20.4
59.00	5349.6	12.6	92.00	4869.5	16.6	125.00	4258.4	20.6
60.00	5337.0	12.7	93.00	4852.9	16.7	126.00	4237.8	20.7
61.00	5324.3	12.9	94.00	4836.2	16.8	127.00	4217.1	20.8
62.00	5311.4	13.0	95.00	4819.4	17.0	128.00	4196.3	20.9
63.00	5298.4	13.1	96.00	4802.4	17.1	129.00	4175.4	21.0
64.00	5285.3	13.2	97.00	4785.3	17.2	130.00	4154.4	21.2
65.00	5272.1	13.3	98.00	4768.1	17.3	131.00	4133.2	21.3
66.00	5258.8	13.5	99.00	4750.8	17.4	132.00	4111.9	21.4
67.00	5245.3	13.6	100.00	4733.4	17.6	133.00	4090.5	21.5
68.00	5231.7	13.7	101.00	4715.8	17.7	134.00	4069.0	21.6
69.00	5218.0	13.8	102.00	4698.1	17.8	135.00	4047.4	21.8
70.00	5204.2	13.9	103.00	4680.3	17.9	136.00	4025.6	21.9
71.00	5190.3	14.1	104.00	4662.4	18.0	137.00	4003.7	22.0
72.00	5176.2	14.2	105.00	4644.4	18.2	138.00	3981.7	22.1
73.00	5162.0	14.3	106.00	4626.2	18.3	139.00	3959.6	22.2
74.00	5147.7	14.4	107.00	4607.9	18.4	140.00	3937.4	22.4
75.00	5133.3	14.5	108.00	4589.5	18.5	141.00	3915.0	22.5
76.00	5118.8	14.7	109.00	4571.0	18.6	142.00	3892.5	22.6
77.00	5104.1	14.8	110.00	4552.4	18.8	143.00	3869.9	22.7
78.00	5089.3	14.9	111.00	4533.6	18.9	144.00	3847.2	22.8
79.00	5074.4	15.0	112.00	4514.7	19.0	145.00	3824.4	23.0
80.00	5059.4	15.2	113.00	4495.7	19.1	146.00	3801.4	23.1
81.00	5044.2	15.3	114.00	4476.6	19.2	147.00	3778.3	23.2
82.00	5028.9	15.4	115.00	4457.4	19.4	148.00	3755.1	23.3
83.00	5013.5	15.5	116.00	4438.0	19.5	149.00	3731.8	23.4
84.00	4998.0	15.6	117.00	4418.5	19.6	150.00	3708.4	23.6
85.00	4982.4	15.8	118.00	4398.9	19.7	151.00	3684.8	23.7
86.00	4966.6	15.9	119.00	4379.2	19.8	152.00	3661.1	
87.00	4950.7	16.0	120.00	4359.4	20.0			

APPENDIX B

SAMPLE CALCULATION OF CALIBRATION DATA FOR THERMOCOUPLES

A sample calculation of the calibration data for the thermocouples is given in this appendix. For this illustration, data taken during Run No. 7-12 shown in Table 11 are used. These data were taken at a temperature of approximately 78.2°K, and the temperature drift was approximately 0.0009 degree per minute. The cryostat was arranged as described in Chapter IV. The system was under high vacuum (1×10^{-6} mm Hg) after warming from temperatures below the fixed point.

The calculation of calibration data for the thermocouples from the experimental data was carried out by one of two methods, the selection of the method used being dependent upon the drift rate of the temperature observed during the run. The first method was extremely simple and consisted of averaging the experimental data obtained for the temperature and e.m.f. The drift rate for data calculated by this method was 0.0005 degree per minute or less. Such a small drift rate was usually obtained only during the runs made at the fixed point temperature, because the system was then essentially at the temperature of the refrigerant.

The second method involved the plotting of temperature versus time for the platinum resistance thermometer, and the interpolation of temperatures at the time e.m.f. measurements were made for the thermocouples. A plot of e.m.f. versus the corresponding temperature was then

Table 11

Sample Calibration Data Sheet, Data for Run No. 7-12

Time A.M.	Resistance, ohms of Th2	Temperature, °K of Th2	e.m.f., microvolts of 100 ohm standard	of T1	of T2	of TC3	of TC4	of TC5
10:19:40	5.94749	78.258						
24:30			103876					
26:00					64794			
27:00				62791				
28:00						5084.7		
28:30								5085.1
29:00							5086.1	
32:20	5.94893	78.269						
35:00			103874					
37:00					64790			
40:00				62787				
41:30						5084.7		
42:00								5085.0
42:30							5086.0	
45:20	5.94993	78.277						
48:00			103875					
49:00					64787			
50:00				62785				
51:00						5084.3		
51:30								5084.6
52:00							5085.8	
54:00	5.95147	78.288						
59:00			103872					
11:00:00					64782			
01:00				62781				
02:00						5084.0		
02:30								5084.3
03:00							5085.3	
06:00	5.95277	78.298						

made to determine the sensitivity of the thermocouples. The determined sensitivity was then used to extrapolate the data to an arbitrarily selected temperature within the data. The e.m.f. data were then averaged to obtain a value at this selected temperature. This average value of the e.m.f. was used as the experimental value of the e.m.f. of the thermocouple at this temperature.

Temperature of platinum resistance thermometer.--In Table 11, resistance and corresponding temperature data for the platinum resistance thermometer are shown. No discussion will be given for the calculation of temperature from the resistance recorded during the run. An outline of this calculation for the range, 20° to 90°K and 90° to 273°K, is given in the Research Notes of Project 116[1] along with the necessary tables and correction plots for making the calculation.

Temperature of thermocouples at any time.--The temperature of the thermocouples at the time the e.m.f. measurements were made was determined from a plot of the temperature versus time for the platinum resistance thermometer (see Figure 10). This assumed that the temperature of the thermocouples was the same as the temperature of the platinum resistance thermometer under the steady-state conditions that existed during the run. Using the data of Table 11 to plot Figure 10, the temperature of the thermocouples at the time e.m.f. measurements were made was read and are tabulated in Table 12.

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[1]Research Notes of Project 116; Low Temperature Laboratory; Engineering Experiment Station; Georgia Institute of Technology; October, 1953 to February, 1955; pp. 15-16, 32, 68-75, 84.

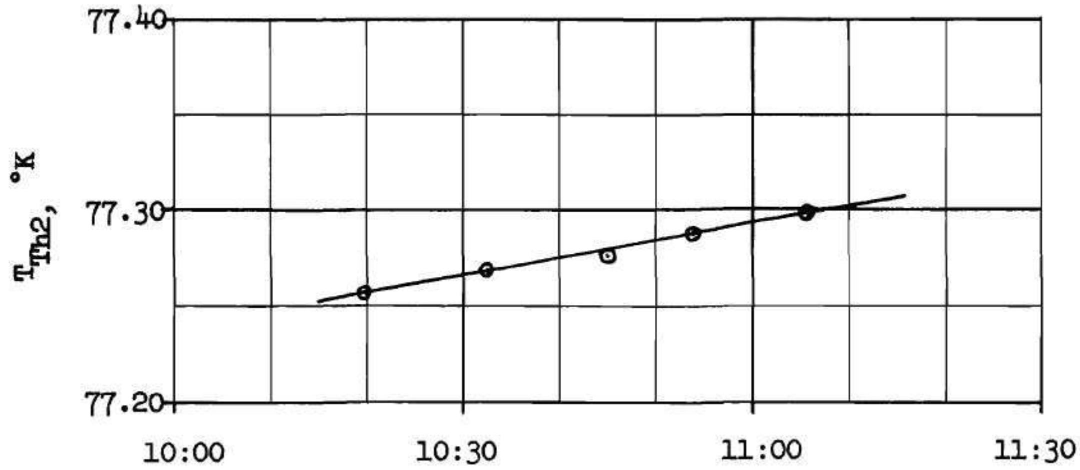

Figure 10

Temperature versus Time
for
Platinum Resistance Thermometer

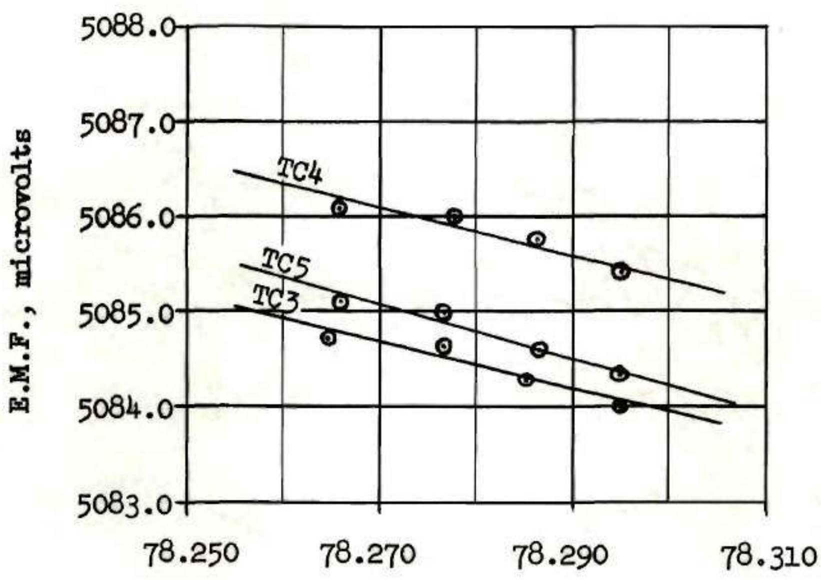

Figure 11

E.M.F. versus Temperature
for
Thermocouples

Table 12

Temperature and E.M.F. Data of Run No. 7-12 for Thermocouples

Time A.M.	Temperature, °K	e.m.f., μv. TC3	e.m.f., μv. TC4	e.m.f., μv. TC5
10:28:00	78.265	5084.7		
28:30	78.266			5085.1
29:00	78.266		5086.1	
41:30	78.277	5084.7		
42:00	78.277			5085.0
42:30	78.278		5086.0	
51:00	78.285	5084.3		
51:30	78.285			5084.6
52:00	78.286		5085.8	
11:02:00	78.295	5084.0		
02:30	78.295			5084.3
03:00	78.295		5085.3	

Sensitivity of thermocouples.--The sensitivity of the thermocouples at any temperature point was determined by evaluating the slope of the e.m.f. versus temperature curve for the thermocouple. The data for the thermocouples shown in Table 12 are plotted in Figure 11. From this figure the following data for TC3 were read to compute the slope of this curve: (1) at $T = 78.261°K$, e.m.f. = $5084.9\,\mu v$. and (2) at $T = 78.299°K$, e.m.f. = $5084.0\,\mu v$. The slope then is equal to:

$$\frac{\Delta \text{e.m.f.}}{\Delta T} = \frac{5084.9 - 5084.0}{78.261 - 78.299} = -\,24\,\mu v/°K$$

The minus sign indicates that the system was heating during the run, and is not significant as far as the sensitivity of the thermocouple is concerned. Thus the sensitivity of the thermocouple TC3 was $24\,\mu v/°K$ at this temperature.

Extrapolation of e.m.f. data to one temperature.--To extrapolate the e.m.f. data to one temperature, the sensitivity of the thermocouple was used to compute a correction to the e.m.f. data at the different temperatures. Also, one temperature within the data was arbitrarily selected as the base temperature (T_b). This correction was computed by the expression:

$$C = s(T - T_b)$$

where C = correction in μv.
s = sensitivity in $\mu v./°K$
T = any temperature in $°K$
T_b = base temperature in $°K$

The correction was then added to the e.m.f. at temperature (T) to obtain the e.m.f. at the base temperature (T_b). For this run, assume the base temperature is 78.265°K, and the sensitivity was 24 μv./°K as previously calculated. Then for example, at a temperature (T) equal to 78.295°K, the correction (C) is:

$$C = 24\ (78.295 - 78.265) = 0.7 \pm 0.44\,\mu v.$$

This correction (0.7 μv.) added to the e.m.f. at 78.295°K (5084.0 μv.) gives an extrapolated e.m.f. at 78.265°K of 5084.7 μv.

Table 13 is a summary of the corrections made to the e.m.f. data at temperature (T) to obtain extrapolated e.m.f. data at the base temperature (78.265°K). These data are for Run No. 7-12 and thermocouple TC3.

Table 13

Calibration Data at 78.265°K
for
TC3 During Run No. 7-12

Temperature, °K	e.m.f., μv. at T	Correction to e.m.f. at T, μv.	e.m.f., μv. at T_b
78.265	5084.7	0.0	5084.7
78.277	5084.7	0.3 ± 0.19	5085.0
78.285	5084.3	0.5 ± 0.32	5084.8
78.295	5084.0	0.7 ± 0.44	5084.7

Value of e.m.f. at base temperature.--The final value of the e.m.f. at the base temperature is computed by averaging the e.m.f. data extrapolated to this temperature. Thus if the four e.m.f. values in Table 13 are averaged, an average e.m.f. of 5084.8 μv. at 78.265°K is obtained for thermocouple TC3.

Data for thermocouples TC4 and TC5.--The experimental data obtained during Run No. 7-12 for thermocouples TC4 and TC5 are shown in Table 11. The e.m.f.-temperature curves for calculating the sensitivity of these thermocouples are also shown in Figure 11. Using the method of calculation outlined for thermocouple TC3, the e.m.f. at 78.265°K for TC4 and TC5 were calculated to be 5086.2 μv. and 5085.2 μv., respectively.

APPENDIX C

CALCULATION OF THERMAL CONDUCTIVITY

The basic relationship for determining the thermal conductivity was presented in Chapter I as equation (2). This relationship is

$$k_m = - \frac{Q}{A \frac{\Delta T}{\Delta x}} \qquad (2)$$

where k_m is the thermal conductivity at the arithmetic mean temperature; Q is the rate of heat flow through the specimen; A is the cross-sectional area of the specimen; and $-\frac{\Delta T}{\Delta x}$ is the temperature gradient along the specimen or the ratio of the temperature difference between two points a known distance apart on the specimen. However, because of the fact that a slight temperature drift was always present, it was necessary to modify equation (2) to take into account the fact that a small amount of heat was actually being stored or lost from the specimen, clamps, and specimen heater block. Knowing the heat capacity of these parts and assuming a constant drift rate in the temperature, the fundamental differential equation of heat flow was solved to give an expression for the actual heat flow Q through the specimen as represented by equation (7)[1]. It should be noted that when the drift rate $\frac{dt}{d\theta}$ is zero, Q in equation (7) becomes equal to Q_o, the heat generated by the electrical heater.

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[1]The derivation of this correction equation is given by Cooper (14). The derivation assumes that the diffusivity $\beta = k/C_p\rho$ is constant.

$$Q = Q_o \left[1 - \frac{1}{Q_o} \left\{ (m_H + m_{clamp}) \; C_c + (m_{x_1 - x_o} + 1/2 \; m_{x_2 - x_1}) \; C_s \right\} \frac{dT}{d\theta} \right] \quad (7)$$

where,

Q	=	heat entering the specimen at the centerline of the lower clamp, watts
Q_o	=	heat input to specimen heater, watts
m_H	=	mass of copper specimen heater, grams
m_{clamp}	=	mass of lower copper clamp, grams
$m_{x_1 - x_o}$	=	mass of specimen between heater and centerline of lower clamp, grams
$m_{x_2 - x_1}$	=	mass of specimen between centerline of clamps, grams
C_c	=	specific heat of copper, joules/gram-°K
C_s	=	specific heat of specimen, joules/gram-°K
$\frac{dT}{d\theta}$	=	mean drift of thermocouples, °K/sec.

Setting,

$$\delta = \frac{1}{Q_o} \left[(m_H + m_{clamp}) \; C_c + (m_{x_1 - x_o} + 1/2 \; m_{x_2 - x_1}) \; C_s \right] \frac{dT}{d\theta} \quad (8)$$

then, δ is defined as a correction factor applied to the heat input to the specimen heater that depends on the temperature, the specimen, the specific heat of the specimen and copper at the temperature, the drift rate, and the heat input to the specimen heater.

The drift rate in equation (8) is expressed in degrees per second, which may be expressed in microvolts per hour since

$$\frac{dT}{d\theta} = \frac{1}{3600 \; \frac{dE}{dT}} \left(\frac{dE}{d\theta}\right) \quad (9)$$

where,

$\frac{dT}{d\theta}$ = mean drift rate, degree/second

$\frac{dE}{dT}$ = sensitivity of thermocouples at the temperature, microvolts/degree

$\frac{dE}{d\theta}$ = mean drift rate, microvolts/hour

$\frac{1}{3600}$ = conversion factor, hours/second

Substituting equation (9) into equation (8), it follows that

$$\delta = \frac{1}{Q_o}\left[(m_H + m_{clamp})\ C_c + (m_{x_1} - x_o + 1/2\ m_{x_2} - x_1)\ C_s\right]\left(\frac{1}{3600\ \frac{dE}{dT}}\right)\frac{dE}{d\theta} \quad (10)$$

In equation (10), the specific heat of copper and the specimen and the sensitivity of the thermocouples are dependent upon the temperature. To further simplify equation (10), substitute the symbol, α, for these variables and the constant terms in the equation.

Then,

$$\delta = \frac{\alpha}{Q_o}\ \frac{dE}{d\theta} \quad (11)$$

The value of the term α was evaluated in the temperature range 70° to 150°K using the dimensions of the specimen, the heat capacity of copper and the specimen, and the sensitivity of the thermocouple. It was found that over this range α was essentially constant and was equal to 2.90×10^{-4} watt-hour per microvolts. Using this value for α a plot of δ versus $\frac{dE}{d\theta}$ was made for various values of Q_o (see Figure 12). This figure then permits one to determine the correction factor, δ,

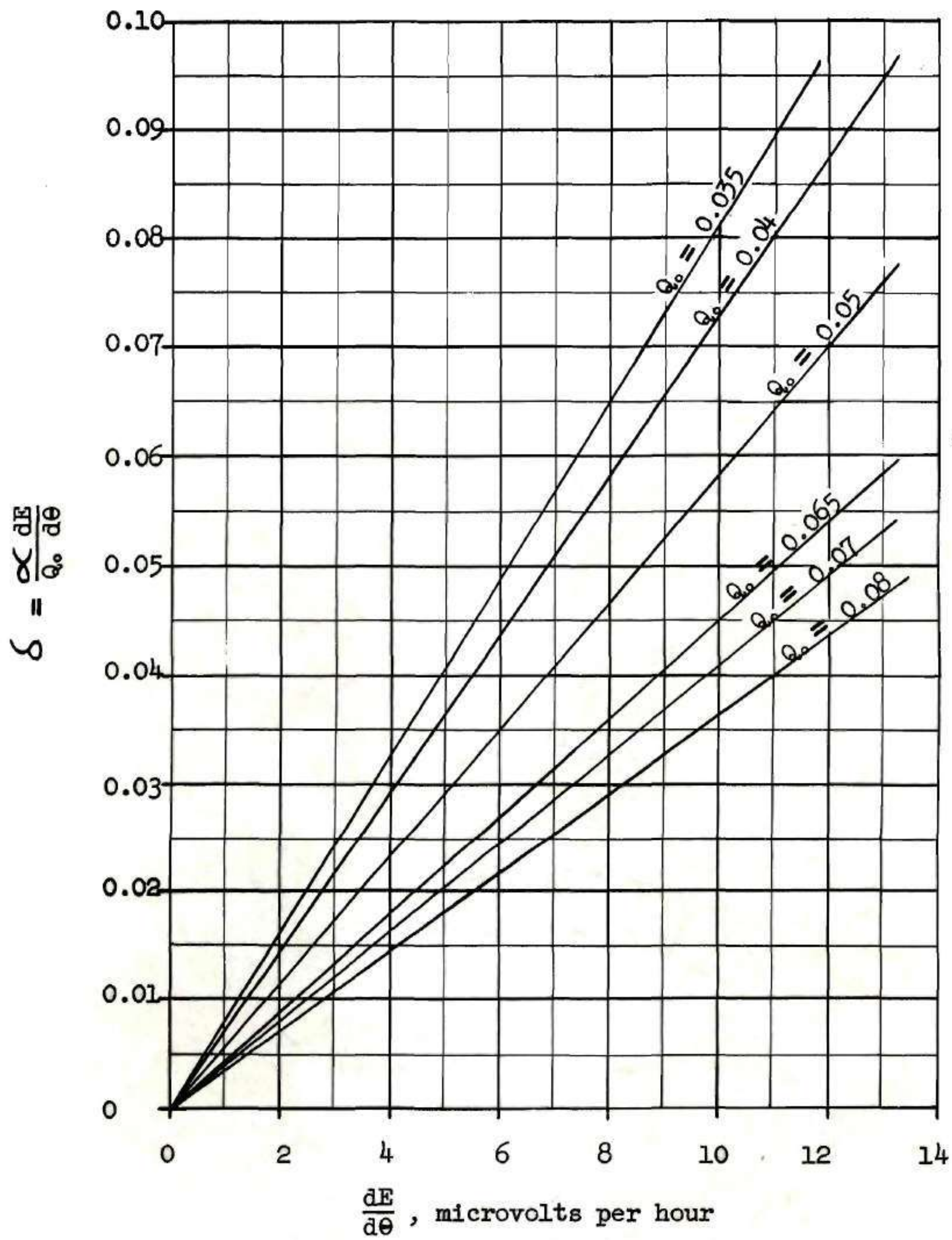

Figure 12

Correction to Heat Input

rapidly from a knowledge of the drift rate and the heat input to the specimen heater.

It follows from equations (7) and (8), that the heat flowing through the specimen may be expressed as,

$$Q = Q_o \left(1 - \delta\right) \tag{12}$$

Substituting equation (12) into equation (2), the final expression used to calculate the thermal conductivity is,

$$k_m = - \frac{Q_o \left(1 - \delta\right)}{A \frac{\Delta T}{\Delta x}} \tag{13}$$

Equation (13) assumes that there is no loss of heat from the heater due to radiation and to conduction through the specimen heater leads and thermocouple leads. Based on the work of previous workers with this type of equipment this assumption is not entirely valid even at these low temperatures. Time did not permit an experimental investigation of these losses in this work. Calculations indicated, however, that both radiation and conduction losses were expected to be small.

The terms, A and Δx, in equation (13) were evaluated before making the experimental measurements. These measurements were made at room temperature. Four measurements along the specimen were made with a micrometer, and the measured diameter was 0.250 ± 0.0005 inches (0.635 cm). The cross-sectional area, A, of the specimen normal to the flow of heat was 0.3167 sq cm. Measurements were also made with a micrometer to determine the distance between the centerlines of the two copper clamps on which the thermocouples were attached. These measurements gave an average distance, Δx, of 5.030 $\pm$ 0.005 inches (12.78 $\pm$ 0.013 cm).

For the completion of this discussion on the calculation of the thermal conductivity, data for the temperature difference, ΔT, and the heat input to the specimen heater, Q_o, will be evaluated from Table 14. This table shows a sample sheet of experimental data taken at steady-state conditions during Run No. 1. As will be later shown, the mean temperature of the run was 88.73°K, and the mean drift rate of the thermocouples was 2.0 microvolts per hour.

The heater power (Q_o) was calculated from the potential data measured for the standard resistance and the volt box. The following steps are included in this calculation.

All volt box readings were made with the volt box in the ratio of 1 to 200, which required that these potential values be multiplied by 200 to get the true heater potential.

$$E_h = 200\ E_{vb} = 200\ (0.016353)$$

$$= 3.2706 \text{ volts}$$

Since the 1-ohm standard resistance was in series with the heater, the current could be calculated and is:

$$I_s = \frac{E_s}{R_s} = \frac{0.021051 \text{ volts}}{1.00044 \text{ ohms}}$$

$$= 0.021042 \text{ amperes}$$

A small fraction of the current passed through the volt box, which was essentially a 60,000 ohm resistor in parallel with the specimen heater.

Table 14

Sample Sheet of Experimental Data

for Thermal Conductivity Determination

Data of Run No. 1

Time		e.m.f., microvolts
7:36:00 P.M.	TC3	4967.0
37:00	TC4	4877.9
38:00	Volt box	16353
40:00	1-ohm Standard	21051
41:30	TC4	4877.9
42:30	TC3	4967.0
8:07:00	TC3	4966.1
08:00	TC4	4876.8
09:00	Volt box	16356
10:00	1-ohm Standard	21054
11:00	TC4	4876.7
12:00	TC3	4966.1

This volt box current could be represented:

$$I_{vb} = \frac{200\ E_{vb}}{R_{vb}} = \frac{200\ (0.016353)\ \text{volts}}{60{,}000\ \text{ohms}}$$

$$= 0.000054\ \text{amperes}$$

Subtracting the volt box current from the total current, the current through the heater is:

$$I_h = I_s - I_{vb} = 0.021042 - 0.000054$$

$$= 0.020988\ \text{amperes}$$

The heater power is given by the expression,

$$W_h = I_h\ E_h = (0.020988\ \text{amperes})\ (3.2706\ \text{volts})$$

$$= 0.06864\ \text{watts}$$

and represents the heat input (Q_o) to the specimen heater.

The correction factor, δ , which must be applied to the heat input (Q_o) in equation 13 because of temperature drift, was read from Figure 11 by interpolation between the $Q_o = 0.065$ and $Q_o = 0.07$ lines and for a mean drift rate of 2.0 microvolts per hour. This factor is 0.007.

The e.m.f. data for the thermocouples were converted to a temperature by linear interpolation in Table 10, Appendix A. Thus the temperatures of TC3 and TC4 were 85.97°K and 91.49°K, respectively, at the time the data above were taken. The mean temperature of this run was 88.73°K, and the temperature difference (ΔT) to be used in equation 13 was 5.52°K. Substituting the values of A, Δx, Q_o, δ, and ΔT into equation 13,

$$k_m = \frac{(0.06864 \text{ watts})\ (1 - 0.007)}{(0.3167 \text{ sq cm})\ \frac{(5.52°K)}{(12.781 \text{ cm})}}$$

$$= 0.498 \text{ watts/cm - °K}$$

Thus the mean thermal conductivity of this brass specimen was 0.498 watts per cm - °K at a mean temperature of 88.73°K.

Using this method, the thermal conductivity was calculated for each experimental run. The results are shown in Table 9, Chapter V.

APPENDIX D

CALIBRATION OF CARBON RESISTANCE THERMOMETERS

The carbon resistance thermometers and the method of mounting them are described in Chapter II. The electrical circuit used is described in Chapter III. The procedure for the calibration of the carbon resistance thermometers was discussed in Chapter IV with the procedure for the calibration of the thermocouples. In this appendix a brief discussion is given on the calculation and results of the data obtained for the carbon thermometers.

Calculation of data.--The first step in the calculation was to determine the resistance of the carbon thermometers, since this was not measured directly with the measuring instruments. The resistance was easily calculated from the experimental data for the e.m.f. across the carbon resistors and the e.m.f. across the standard resistance, which was in series with the thermometers. Using Ohm's law, the current through the circuit was calculated from the e.m.f. across the standard resistance and the known resistance of the standard resistance. Then by assuming that there was no change in the current with time, the resistance of the carbon thermometer was calculated by substituting into Ohm's law the e.m.f. across the thermometers and the current through the circuit. For example, in Table 11, Appendix B at 10:26:00 A.M., the e.m.f. across the standard resistance was 103876 microvolts. Thus the current through the standard resistance and the circuit was 0.00103876 ampere. Also in

Table 11, Appendix B the e.m.f. across thermometer T1 was 62791 microvolts at 10:27:00 A.M. Assuming the current to be the same at 10:27:00 A.M. as it was at 10:26:00 A.M., the resistance of the thermometer T1 was 60.4480 ohms. Using this method the resistance of the carbon thermometers was calculated at the time e.m.f. measurements of the thermometers were made.

After the resistance of the carbon thermometers was computed, the second step of the calculation was to compute an average resistance at each temperature point of the data. This calculation was made in the same manner as discussed for the thermocouples in Appendix B. Thus by changing the phrase "the e.m.f. of the thermocouples" to read "the resistance of the carbon thermometers", the discussion in Appendix B can be applied to the carbon thermometers. Using the data shown in Table 11, Appendix B, the average resistances of the thermometers T1 and T2 were then calculated to be 60.4490 and 62.3762 ohms, respectively, at 78.263°K.

<u>Results of calibration</u>.--The calibration data for the carbon thermometers over the range 55° to 150°K were evaluated after Runs No. 2 and 4. The evaluation of these data was based on the data taken at the fixed point, because this point represented the temperature for which the best calibration data were obtained. A summary of the calibration data at the fixed point is shown in Table 15.

The data of Run No. 4 in Table 15 were plotted, and a straight line, which fitted the data to $\pm$ 0.02 ohm, was drawn through the data for each thermometer. When the data of Run No. 2 were plotted with the

Table 15

Summary of Calibration Data At Fixed Point for Carbon Resistance Thermometers

Run No.	Point No.	Temperature,(1) °K	Average Resistance, ohms T1	T2
5	5-2	77.604	60.726	62.706
6	6-1	77.670	60.613	62.573
6	6-7	77.948	60.508	62.455
4	4-1	77.987	60.979	63.033
7	7-1	78.056	60.547	62.478
7	7-6	No Data Taken		
7	7-12	78.263	60.449	62.376
5	5-1	78.250	60.538	62.514
5	5-8	78.356	60.467	62.420
2	2-1	78.499	61.100	63.256
4	4-12	78.762	60.712	62.729
5	5-10	79.090	60.248	62.194
4	4-8	79.597	60.484	62.507

(1) Temperature of platinum resistance thermometer.

data of Run No. 4, these data deviated from the lines through the data of Run No. 4 by + 0.30 and + 0.37 ohm for T1 and T2, respectively. This deviation of + 0.30 and + 0.37 ohm is equivalent to about one degree in temperature. Because of the deviation between the data of these two runs, it was concluded that it was necessary to calibrate the carbon thermometers each time they were to be used as thermometers.

The slope of the lines through the data for each carbon thermometer represented the sensitivity of the thermometer at the fixed point. Thus the sensitivity of T1 and T2 was determined to be 0.30 and 0.32 ohms per degree, respectively, which was 0.5 per cent per degree for each thermometer. At this same temperature, the sensitivity of the copper-constantan thermocouples was 0.3 per cent per degree. Thus even though the data indicated that the carbon resistance thermometer was a more sensitive device for measuring temperature, it was not used because of its non-reproducibility. Consequently, calibration data for the carbon thermometers were taken only at the fixed point in Runs No. 5, 6, and 7. This was done to see if a reproducible resistance could be obtained in later calibration runs.

The data of Run No. 5 when plotted with the data of Runs No. 2 and 4 deviated from the lines through the data of Run No. 4 by - 0.36 and - 0.48 ohms for T1 and T2, respectively. This indicates that the above conclusion on the reproducibility of the thermometers was true. Also, since between each of these runs the thermometers were cooled from room temperature, it suggested that cooling from room temperature was in some way effecting the carbon resistors.

When the data of Runs No. 6 and 7 were added to this plot, the above conclusion on the reproducibility of the resistance failed. The data of Run No. 6 agreed with the data of Run No. 7 to about - 0.04 ohms, which is equivalent to about 0.1 degree. Also, the data of these runs agreed with the data of Run No. 5 to about - 0.10 ohms or 0.3 degree. It was also noted that the data for Run No. 7 fell between the data of Runs No. 5 and 6. These facts then indicated that the resistance of the carbon thermometers was reproducible within limits of less than one degree in temperature. They also indicated that the carbon thermometers were affected by cooling from room temperature, but that after cooling from room temperature a few times, the cooling effects were stabilized. Thus a reproducible resistance was obtainable. As a result of these facts straight lines were drawn through the data of Runs No. 5, 6, and 7 for each thermometer. Each line fitted the data for the thermometers to within $\pm$ 0.06 ohms. The slope of these lines e.g. the sensitivity of the thermometers was 0.30 and 0.32 ohms per degree for T1 and T2, respectively, and agreed with the sensitivity as previously determined. Using the sensitivity, the data of these runs for each thermometer were extrapolated to a temperature of 78.250°K. The average resistance and the average deviation of the carbon thermometers were 60.48 $\pm$ 0.04 and 62.43 $\pm$ 0.05 ohms for T1 and T2, respectively, at this temperature. This deviation is equivalent to $\pm$ 0.15 degrees of temperature.

Accuracy of results.--The accuracy of the calibration data at the fixed point was effected by the reproducibility of the resistance of the carbon thermometers, the precision of the measurements of e.m.f. and temperature data, and the precision of the instruments. Considering all these factors it is believed that the accuracy of the calibration data is ± 0.22 degrees for both thermometers.

Conclusions.--The following conclusions are based on the results of the calibration of the carbon resistance thermometers:

(1) The carbon resistance thermometers are more sensitive devices for measuring temperature than the copper-constantan thermocouples.

(2) The resistance of the carbon thermometers was reproducible to ± 0.05 ohm (± 0.15 degree) at a temperature of 78.25°K.

(3) Cooling the carbon thermometers from room temperature to 78°K affected the resistance of the thermometers, but these cooling effects appeared to be eliminated after cooling from room temperature several times.

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